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DEVELOPMENT OF A HAZARD CLASSIFICATION PROCEDURE FOR INPROCESS PROPELLANT AND EXPLOSIVE MATERIALS

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A hazards classification procedure has been developed for inprocess propellant and explosive materials. In developing the procedure, accident reports in the DOD Explosives Safety Board files, hazards analyses, and existing test methods were reviewed. These were used as the basis for developing a preliminary structure for the hazards classification procedure. The most promising test for incorporation into the procedure was experimental evaluations. Evaluations were completed for local impact, rubbing friction, local thermal,

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20. ABSTRACT (continued)

regional thermal, electrostatic discharge, critical diameter, critical layer thickness, tube transition, layer transition, mass explosion, mass fire and firespread tests. The results of these tests served as the basis of the final hazards classification procedure.

FOREWORD

At IITRI E. Swider contributed significantly to all aspects of the program, J. Daley and M. Amor conducted the majority of the testing, R. Joyce and D. Hrdina accomplished the electrostatic test evaluations and contributions to many of the other tests, A. Goldsmith of the Chicago City Collage and T. E. Waterman of IITRI contributed to the thermal aspects of the problem, and W. Abel helped to identify the important aspects of process plant operations.

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SUMMARY

A hazards classification procedure has been developed for chemical mixtures which exist in propellant and explosive manufacturing operations. The approach used in developing the procedure consisted of the following steps. First, a survey was made of process plant accident reports in the DOD Explosives Safety Board files and of available hazards analyses. These were reviewed in order to identify the important aspects of the problem (i.e., ignition modes, stimulus intensities and consequences). A survey of existing potentially applicable tests was also conducted to identify the techniques previously used. A preliminary procedure was then formulated and the most promising tests required by the procedure were evaluated experimentally. The evaluations included tests to characterize local impact initiation, rubbing friction initiation, local thermal initiation, regional thermal initiation, electrostatic discharge, critical diameter, critical layer thickness, tube transition, layer transition, mass explosion, mass fire, and firespread. Tests were conducted on four inprocess sample materials (M30 pellets, M1 strands, M26 paste, and RDX slurry). The test results were used to scrutinize the preliminary procedure and identify necessary procedure modifications. The hazards classification procedure was then finalized based on the changes indicated. The final procedure consists of two parts: (1) a sensitivity evaluation to indicate the likelihood of an initiation occurring and identify the dominant stimulus types and (2) an effects evaluation to identify the probable consequence of an initiation and its severity. Based on the effects evaluation, the material is assigned to be a classification in a scheme very similar to the existing NATO-UN system.

BACKGROUND

Objective

The objective of the investigation described in this report is to develop a hazard classification procedure for chemical mixtures which exist in propellant and explosive manufacturing operations. The procedure developed is to form the basis of a regulatory guide to modify or supplement the existing "explosives hazard classification procedures," Department of the Army Technical Bulletin 700-2 (Ref. 1). The existing procedures specifically do not address hazards which exist "during various stages of manufacture and assembly." The procedure developed under the present work is intended to fill that void.

The existing hazard classification procedure (TB 700-2) addresses hazards associated with final product explosives in transport and storage, rather than material forms which exist during manufacture inside process plants. Such inprocess materials exist in a wide variety of material forms (solids, powders, flakes, grains/cylinders, strands, slurries, liquids, emulsions, vapor-air or dust-air mixtures, etc.). These materials are acted on by a wide variety of normal and abnormal operation stimuli in a wide variety of process operations. If an ignition occurs, the result may be anything from a minor reaction which does not propagate to a massive explosion. Other hazards such as toxic gas production also exist, but are not addressed here. An effective hazards classification procedure must address each of these factors in a realistic manner.

Previous Program

This report presents the results of the second project conducted by IIT Research Institute to accomplish the stated objective. The accomplishments of the initial program (Ref. 2) included:

1. A survey of existing hazard classification schemes was made. Most of the existing schemes were found to have distinct weaknesses. The NATO-UN system (Ref. 3) seems to minimize these weaknesses and was tentatively selected as the basis for developing the inprocess hazards classification procedure.

2. The Department of Defense Explosives Safety Board (DDESB) accident records were reviewed to help clarify the hazards which have existed in process operations historically. This includes both the accident consequences and probable causes. A statistical analysis of the collected data was conducted in order to help define the minimum stimulus energy which had to be present in the accidents to have caused initiation, assuming the accident reports cited the correct causes for the accidents. Only

a sampling of the DDESB file was reviewed; the data base for statistical analysis was quite small in many cases; it was suggested that a more thorough search be conducted in the follow-on effort.

3. A preliminary survey was made of existing (primarily sensitivity) tests, and the most promising tests for incorporation into a hazards classification procedure were chosen. Selected tests were experimentally evaluated using four inprocess materials as test samples. These sample materials were:

   a. RDX-H_2O slurry to represent a conveying operation,
   b. M30 pellets to represent a drying operation,
   c. M26 paste to represent a mixing operation, and
   d. M1 strands to represent an extrusion operation.

   The test evaluations included drop weight impact, strip friction, electrostatic discharge (ESD), differential scanning calorimetry (DSC), critical diameter and critical height. It was concluded that for the standard impact, friction and ESD tests, in many cases the sample material form had to be severely altered from the actual inprocess form in order to conduct the test. This could lead to very unrealistic conclusions about the material's sensitivity. DSC appeared to adequately characterize the material's sensitivity to ignition by a regional thermal stimulus.

4. Based on the results of the investigations in the initial program, a preliminary hazards classification procedure was drafted.

In general, the initial work helped to "define the problem." Classification schemes were identified and the NATO-UN scheme was selected as the most promising. Accident consequences, accident causes, and ignition stimuli levels were identified based on a preliminary review of the DDESB accident file, but a more thorough review was needed. Existing test methods were reviewed and selected. These tests were experimentally evaluated, but the need for some modifications of existing tests was pointed out. Very few existing tests could be applied directly to realistically characterize the hazards of inprocess materials. A "first iteration" procedure was drafted but after scrutinizing that procedure format in the intial steps of the follow-on program, the procedure was found to embody several deficiencies and a fresh look was needed.

Program Approach

The first task accomplished on the present program was to critically review the work accomplished under the previous project. Although a sound base was provided, many of the accomplished tasks required expansion. The following program approach was taken in order to finalize the development of a hazard classification procedure for inprocess propellant and explosive materials:

Task 1: Historical Accident Survey

The relevant process plant accident reports in the DDESB file not reviewed in the previous program were collected, reviewed, and added to the reports which were previously collected. The survey of the DDESB accident reports was conducted to determine what types of accidents have occurred historically and what stimuli were felt to be the causes. In addition, by knowing what material was initiated and which stimulus was the most likely cause, the minimum stimulus energy level which had to be present could be estimated in many cases. This helped to "define our problem" by pointing out what consequences, ignition modes, and stimulus intensities have to be represented by the procedure developed.

Task 2: Engineering Analysis Survey

To supplement the information sought under Task 1, hazards analyses conducted for process plants were also reviewed. Stimulus types uncovered by the hazards analyses and stimulus energy levels ("inprocess potentials") estimated from hazards analysis engineering analyses were summarized in the same manner as was done for the DDESB historical data. By combining the results of Tasks 1 and 2, approximate intensities of each stimulus type for each type of process operation were estimated. These values were later used to help define the significance of sensitivity test results.

Task 3: Survey of Existing Tests

The survey of test methods which was conducted on the previous program was expanded and used to develop a list of tests which might be applicable to hazards classification of inprocess materials. This also showed which phenomena previous investigators felt were important and how they felt these phenomena could be characterized in tests.

Task 4: Define Classification Procedure Structure

Tasks 1, 2 and 3 showed which accident consequences are of major concern, which stimuli are most important in causing the accidents, and what tests have been used to characterize these hazards. With this information, several options were seen to exist which might be used to classify the hazards of inprocess materials. After deciding to use the procedure to assign inprocess materials to categories, the same or nearly the same as the NATO-UN categories a preliminary procedure structure was formulated. During the program, the procedure structure went through many iterations before being finalized.

Task 5: Select Candidate Classification Tests and Evaluate

Based on the procedure structure developed under Task 4, candidate tests were chosen for experimental evaluation. The same four sample materials used during the initial project were used again to

experimentally evaluate the tests selected for hazards classification. In many cases, the initial form of the test had to be modified one or more times before settling on a test felt to be suitable for the procedure.

Task 6: "Validate" Procedure

Using the test data for the four sample materials (where test evaluations were done) and some sensitivity test data from the literature (where test evaluations were not done), the classification procedure was exercised. The four sample materials were classified using the procedure. It is not felt that this is a full validation of the procedure. A much more extensive validation of the procedure for a wide variety of material forms, and for some materials with an accident history, is strongly suggested.

Task 7: Finalize Procedure

After preliminarily "validating" the procedure, the procedure was finalized. The final procedure is presented in Appendix E of this report.

As background, some discussion will be given to the philosophy behind the structure of the hazard classification procedure which has evolved out of this program. As mentioned previously, it was decided early in the program to have the procedure structured to assign inprocess materials to categories in the NATO-UN classification scheme or a scheme very much like that one. The NATO-UN system seems to minimize the weaknesses noted in Reference 2 for the different classification systems. The NATO-UN scheme is based on the consequences of an initiation and is used to specify quantity-distance requirements. The hazards which exist in a process plant are actually related to both the consequences and the likelihood of the consequences occurring. In this sense, hazards classification should be more of a risk evaluation. Table 1 outlines three of the more obvious bases which could be used to identify an appropriate hazards class. The procedure should yield a classification which not only identifies the worst possible consequences (i.e., quantity-distance) but a (perhaps separate) number should also be assigned which indicates how likely the consequence is to occur. This second number would be based on sensitivity testing whereas the consequence class (giving quantity-distance) would be based on the effects testing. Critical dimension and transition testing helps to identify what the worse credible consequence is and thus which effects tests should be conducted. The procedure developed under this program emphasizes classification by the consequence (i.e., the NATO-UN system) but also considers sensitivity.

In the sections which follow, each of the outlined program tasks will be discussed in greater detail. The final procedure will be presented with some discussion of the options which were considered. The "validation" of the procedure will be discussed, and conclusions and recommendations for further work will be delineated.

Table 1

Possible criteria for classifying inprocess materials

1. Classify Based on the Consequence, e.g.:

   - airblast/fragments
   - fireball
   - sustained mass fire
   - firespread
   - minor consequence

   TELLS: Safe Separation Distance and/or Required Structural Strength, Safety Features, etc.

2. Classify by Material Sensitivity, e.g.:

   - local impact
   - regional impact
   - impingement
   - rubbing friction
   - local thermal
   - regional thermal
   - electrostatic discharge

   } →

   - easily ignited
   - difficult to ignite
   - will not ignite

   TELLS: How Likely an Ignition is and What Safety Precautions Should be Taken to Minimize the Possibility of an Ignition

3. Classify by Total Risk to Life and/or Structures

   RISK = Likelihood of Ignition (Sensitivity) x Extent of Damage (Consequence)

   TELLS: Safe Separation and/or Structural Safety Design Requirements with an Assessment of the Urgency Based on Likelihood of Occurrence

ANALYSIS OF DDESB ACCIDENT DATA

Early in 1978, IITRI personnel visited the DoD Explosive Safety Board to collect the remaining process plant accident data (data not compiled on the previous hazards classification program). The accident reports which were newly collected were reviewed and tabulated. If an accident report did not specify the most probable initiation stimuli (e.g., most short telephone reports), the probable cause entry on our table was "Unknown; No Specification", and the incident was not used in the statistical analysis. This was different from the statistical analysis of the prior contract, where unknown causes were used as data points for all of the initiation stimuli types.

Next, the accident reports compiled under the previous contract were added to the new list. In many cases this involved recategorizing the incidents into the new categories of process operations. A summary table was developed combining the newly collected data with the incident reports collected under the previous contract. This table is presented in Appendix A. A summary of the incident reports documented in the DDESB file is presented in Table 2. Table 2 lists the different categories of process operations and the number of accident reports in the file which involved each of these operations. Not all of the reports in the DDESB file gave probable cause. For those reports which cited one or more probable cause, the distribution of probable causes for each operation type is given in Table 3.

The accident data was grouped as shown in Table 3, by process operation and ignition stimulus type. For each group, a statistical analysis of the usable data was accomplished to help estimate the different stimulus energy levels which would have had to have been present from the operation (normal operation, off design operation or accidental failure mode) in order for the energetic material present to have been initiated. The method that was used to accomplish this can best be explained using an example. Suppose we are analyzing a fictitious type of process operation known as "spraying". Suppose also that there were seven impact and five thermal "spraying" incidents uncovered in the historical file. When we evaluate the thermal "spraying" cases we find that thermal sensitivity tests were done for only three of the materials involved. Without doing further sensitivity tests at this point in time, we only have three cases which can be used in the statistical analysis. Each of these cases is interpreted in the following way. If in case A, the material involved is known to have a thermal ignition point of 350°C from prior sensitivity testing, we assume that at least 350°C had to be present in the process operation in order for the ignition to have occurred by a thermal stimulus. Therefore, the distribution of energy levels represented by the historical data represents the minimum energy levels which are expected to have been present in order for the materials involved to have been ignited. These data do not always conform to a normal distribution. This was clearly seen by plotting the number of

Table 2

Summary of incident reports documented from DDESB file

Process operation	Number of reports documented	Percent of incident reports in each category
Belt Conveyors	4	0.7
Screw Conveyors	1	-
Bucket Conveyors	0	-
Pneumatic Conveyors	1	-
Hoppers	19	3.3
Tote Bins	1	-
Screening, Sieving, Sifting	15	2.6
Pressing, Cartridging	84	14.5
Extrusion, Rolling	11	1.9
Mills	26	4.5
Glazing, Coating, Batch Drum Operations	6	1.0
Drying, Dry House, Oven	63	10.9
Melt Pour, Casting	25	4.3
Chutes	0	-
Reactors	32	5.5
Washing	5	0.9
Mixing	74	12.8
Gravity Separators	3	-
Centrifugal Separators	0	-
Product Pumps	6	1.0
Filters	2	-
Flaker Drum, Belt Flaker	0	-
Distillations	3	-
Solvent Recovery	6	1.0
Mix-Melt Mix Operations	0	-
Recystallization	2	-
Neutralizing	2	-
Packaging/Filling (dry materials) Assembly, Loading/Unloading; Transfer	114	19.7
Machining	21	3.6
Maintenance	40	-
Storage	13	-
TOTAL	579	100

Table 3

Summary of incident reports with the probable initiation stimuli specified

Process operation or component	Incident reports with identifiable causal stimuli[a]	Impact	Friction[b]	ESD	Thermal	Thermochemical exothermic reaction	Impingement	Adiabatic compression	Compression pinch	Electrical
Pressing	53	20	40	1	1			10	1	
Extrusion	7	2	6		1			3		
Mixing	41	12	36	5	1	3				1
Filling	75	32	43	27	1		1			
Drying	39	9	7	2	24					1
Melt-pour; casting	16	5	4	2	9					
Nitration; reactor	25	1	3			23				
Machining	20	2	20	1						
Mills	19	3	15	1	1			1		
Screening	10	5	8	4			1			
Glazing	3		1							2
Belt conveyors	3	1	2							
Screw conveyors	1	1	1							
Pneumatic conveyors	1		1							
Pumping	5	2	3		2					
Hoppers	15	5	14	5						
Tote-bin	1	1								
Washing	3	1	2		1	1				
Separators	3	1	1			2				
Distillations	2				2					
Solvent recovery	2		1	1						1
Recrystallization	3	2	2		1					
Neutralizing	2	1	1	2						1
Filter	2		2	2		1				
Maintenance	33	9	19	4	3	2				
Storage	5		2		3					
Totals	389	115	235	57	50	32	2	14	1	6

[a] Generally several stimuli were specified in each report as the probable causes.

[b] Note the incident reports giving friction as a cause do not necessarily correspond to exposure of the process material directly to friction. The exposure in many cases is indirect, such as a drive belt and stuck pulley heating up by friction. In those cases, the direct stimulus is actually thermal.

occurrences at each energy level as a function of energy level. However, by assuming the data conforms to a normal distribution and by computing the mean plus one standard deviation, we have a consistent procedure for selecting a process energy level which should represent a "high inprocess value." If the distribution were normal, the mean energy level (μ) plus one standard deviation (σ) would include about 84 percent of the cases. Two standard deviations would include about 98 percent of the cases and three standard deviations would include about 99.9 percent. Since many of the distributions are not normal, the $\mu + \sigma$ value is merely a consistent technique for selecting an inprocess energy level which should be a relatively high value for a process operation. Figure 1 presents the energy levels derived using the data for all process operations as a single sampling for each stimulus. Bars showing the mean and one standard deviation spread are shown for impact, friction, ESD, thermal and impingement ignition stimuli. Similar bars have also been developed for all the subsets of data (process operations and stimulus types). These are summarized in Table 4.

Two points concerning Table 4 should be noted. First, whenever only a mean stimulus value is shown, this implies that no deviation in the energy level data was present. In other words all the data was at the same level or only one data point was available. Second, in many cases a note is made of the number of incident reports citing other types of stimuli. When these other incidents are mentioned, a statistical analysis for those stimuli could not be accomplished because sensitivity data was not available for the materials involved.

Impact

$\mu = 3.81$
$\sigma = 3.45$

7.26
— 3.81 $\times 10^4 \frac{J}{m^2}$
0.36

$7.26 \times 10^4 \frac{J}{m^2}$

Friction

$\mu = 2.48$
$\sigma = 1.75$

4.23
— 2.48 $\times 10^8 \frac{w}{m^2}$
.73

$4.23 \times 10^8 \frac{w}{m^2}$

ESD

$\mu = 1.06$
$\sigma = 3.32$

4.37 + seems very high
— 1.06 joules
-2.26

4.37 joules

Thermal (not runaway reactions)

$\mu = 328$
$\sigma = 105$

432
— 328 °C
223

432°C

Impingement

$\mu = 152$
$\sigma = 0$

— 152 $\frac{m}{s}$

$152 \frac{m}{s}$

Fig 1 Total sample categorized by stimulus only

Table 4

Summary of statistical analysis of historical data

Process operation	Stimulus type	Mean stimulus level[a]	Mean plus one standard deviation[a]
Melt pour casting	Impact	6.11	9.39
	Friction	2.5	
	ESD	0.5	
	Thermal	327	423
Pressing	Impact	3.54	6.76
	Friction	3.46	5.41
	Also had one thermal, ten adiabatic compression and one compression/pinch cases		
Reactors (all nitrators)	Friction	0.378	0.527
	Thermal	319	415
Extrusion	Impact	1.0	1.0
	Friction	1.68	3.07
	Thermal	167	
	Also had three adiabatic compression cases		
Machining	Impact	0.34	
	Friction	3.37	4.25
	ESD	0.26	
Mixing	Impact	3.48	5.17
	Friction	3.98	6.51
	ESD	0.014 (person charge 0.015)	
	Also one electrical, one thermal hot spot and three thermal exothermic cases		
Drying	Impact	2.79	6.48
	Friction	4.01	
	ESD	0.0084	0.0163
	Thermal	384	481
	And one electrical case		
Washing	Impact	2.2	
	Friction	0.49	0.89
	Thermal	465	

[a]Units for stimuli are: Impact - $J/m_2^2 \times 10^{-4}$
Friction - $w/m^2 \times 10^{-8}$
ESD - joules
Thermal - °C
Impingement - m/s

Table 4

Summary of statistical analysis of historical data (concl)

Process operation	Stimulus type	Mean stimulus level[a]	Mean plus one standard deviation[a]
Maintenance	Impact	4.45	7.63
	Friction	1.15	2.61
	ESD	0.0028 (person charge 0.015)	
	Thermal	249	303
Storage	Thermal	289	384
	Also two friction cases		
Neutralizing	Impact	2.5	
	Friction	0.292	
	ESD	12.5	
	Also one electrical case		
Recrystallization	Impact	6.8	
	Friction	4.29	
	Thermal	216	
Separators	Thermal	222	
	Also one impact and one friction case		
Hoppers	Friction	2.03	3.46
	ESD	1.26	
	Also five impact cases		
Product pump	Impact	8.5	
	Friction	3.59	4.42
	Thermal	250	
Screw conveyors	Impact	2.5	
	Friction	0.97	
Belt conveyors	Friction	0.291	
	Also one impact case		
Filling	Impact	3.63	8.27
	Friction	1.8	3.30
	ESD	0.107	0.45
	Also one thermal and one impingement case		
Screening	Impact	3.11	5.33
	Friction	3.4	4.45
	ESD	0.0063	0.013 (person charge 0.015)
	Impingement	152	
Milling	Impact	1.20	1.49
	Friction	3.22	4.50
	ESD	0.014 (person charge 0.015)	
	Also one adiabatic compression and one thermal case		

ESTIMATES OF INPROCESS STIMULUS ENERGIES FROM HAZARDS ANALYSIS REPORTS

In order to cross-reference the stimulus energies estimated from the historical data and to assure that no credible ignition modes were missed in the historical data, process plant hazards analysis reports readily available at IITRI were reviewed. The reports from which data was collected are listed in Table 5. This list does not by any means include all hazards analyses that have been done. A much more comprehensive survey of hazards analysis reports is likely to improve the accuracy of the inprocess stimulus energies estimated by this technique. The data in the reports was categorized by process operation and stimulus type. Just as was done for the historical data, the mean plus one standard deviation stimulus level was computed for each category. In Table 6, the inprocess energies from the historical data and hazards analysis engineering analyses are summarized. The table shows the mean and mean plus one standard deviation of the ignition energy (or energy related parameter) in each category. Where comparisons between the historical data and engineering analyses could be made, the values were generally comparable. Impact was an exception to this. For impact in "melt pour-casting operation," "wash, mix and hold tanks," and "product pumps and valves," the historical data values were significantly lower than the engineering analysis energies. This in turn influenced the "all operations" category for impact. The friction, ESD, and regional thermal stimuli showed generally good agreement between the historical and engineering analysis values. There was no historical data for the local thermal, impingement, and intermediate scale impact stimuli, so a comparison could not be made in those cases.

The approach taken in selecting the inprocess potentials" in each category was the following. A "typical high" value was desired in each case; therefore the mean plus one standard deviation ($\bar{x} + \sigma$) values were used. Where only one value (historical or engineering analysis) existed, that value was used, at least as a guide. Where two values existed, the higher value was used, unless it was suspicious for some reason. Where no value existed for a specific process operation, the "all operations" value was used. In some cases, these criteria were overridden. For example, in cases where an ungrounded person can be present, the electrostatic discharge energy from the person will be about 17 millijoules and the inprocess potential should be at least that value. Likewise, in areas where welding could occur (either as a gross human error or as an accepted practice) local hot spots from welding sparks should be on the order of about 1000°C (cooled from molten steel at 1493°C). Therefore 1000°C is the minimum value that should be used.

After several iterations using this approach, combining similar categories to increase the data bases for the statistical analyses, and filling in voids using values derived for similar categories, the inprocess potential energies given in Table 7 were arrived at. It is felt

Table 5

List of hazards analysis reports surveyed

Petino, G. J.	"Engineering Analysis of Equipment and Identification at Hazardous Areas in the "L", "K" and "N" buildings Phase II Task II Report Hazards Research Corp. June 1975.
Petino, G. J.	"Engineering Analysis of Equipment and Identification of Hazardous Areas in the "I" Building" Phase II Task I Report Arpil 1974.
Kristoff, F. T. J. DeGiovanni	"A Hazards Analysis study of the continous TNT Manufacturing Plant" Radford Army Ammunition Plant Hercules, Inc. May 1971.
Albaugh, L. R. Hunt, R. G. Walker, W. L.	"Hazard Evaluation of the Sunflower Second Generation Mechanized Roll Complex Allegany Ballistics Laboratory, Hercules, Inc. July 1973.
	"Hazards study of the continous TNT Manufacturing Plant Extension of PE-243 (General Support) Picatiuny Arseral Radford Army Ammunition Plant Radford, Va. October 1, 1973.
DeGiovanni, J. Smith, D.	"Hazards Analysis of a Centrifugal Pump" Hercules Inc. Allegany Ballistics Laboratory for Holston Defense Corporation November 1971.
Carmack, Sam A. Hansard, H.B. III	"Hazards Analysis of the Prototype Continuous Filtration and Wash Process at Building E-1 Holston Defense Corporation Development and Control Report No 75-0016 May 1975.
Asburg, R. L. Evans. J. L. Ragland, R. S.	"Hazard Analysis and Safety Evaluation of Propellant Manufacturing Porcesses", Final Engineering Report on Production Engineering Project PE-406, Radford Army Ammunition Plant Hercules Inc. Radford, VA July 1977.
Morita D. R. Pape, R.	"Hazards Analysis of the Final Design of the Improved Black Powder Process" IITRI Final Report J6329, For ICI United States, December 1975
Pape, R. Joyce, R.	"Electrostatic Hazards Analysis of a Powder Handling facility "IITRI Final Report 8277, April 1977.

Table 6

Comparison of historical accident data with engineering analysis

Process operation	Local impact (J/m^2)				Rubbing friction (w/m^2)			
	Historical		Eng. anal.		Historical		Eng. anal.	
	$\bar{x}$	$\bar{x} + \sigma$	$\bar{x}$	$\bar{x} + \sigma$	$\bar{x}$	$\bar{x} + \sigma$	$\bar{x}$	$\bar{x} + \sigma$
1. Belt Conveyors			1.72×10^4	3.17×10^4	2.9×10^7	2.9×10^7	2.28×10^7	6.52×10^7
2. Bucket Conveyors			4.62×10^4	4.62×10^4				
3. Screw Conveyors	2.5×10^4	2.5×10^4			0.97×10^8	0.97×10^8		
4. Pneumatic Systems			7.7×10^5	2.1×10^6			4.5×10^6	1.08×10^7
5. Hoppers			88	142	2.03×10^8	3.46×10^8	1.04×10^6	1.75×10^6
6. Tote Bins			5.17×10^3	1.62×10^4			1.24×10^7	6.17×10^7
7. Screening	3.11×10^4	5.33×10^4			3.4×10^8	4.45×10^8	2.13×10^7	4.7×10^7
8. Pressing	3.54×10^4	6.76×10^4			3.46×10^8	5.41×10^3		
9. Extrusion, Rolling	1×10^4	1×10^4			1.68×10^8	3.07×10^8		
10. Mills	1.2×10^4	1.48×10^4			3.22×10^8	4.5×10^8		
11. Glazing, Coating, Batch Drum			1.58×10^4	3.08×10^4			1.51×10^7	5.44×10^7
12. Dryer	2.79×10^4	6.48×10^4	1.9×10^4	3.6×10^4	4.01×10^8	4.01×10^8	9.47×10^7	2.84×10^8
13. Melt Pour, Casting	6.11×10^4	9.39×10^4	3.9×10^5	1.2×10^6	2.5×10^8	2.5×10^8	5.52×10^7	1.99×10^8
14. Churns								
15. Reactors			1.82×10^6	4.36×10^6	0.378×10^8	0.527×10^8	1.28×10^9	5.34×10^9
16. Wash, Mix, and Hold Tanks	2.2×10^4	2.2×10^4	9.45×10^5	2.1×10^6	0.49×10^8	0.89×10^8	2.45×10^8	2.45×10^8
17. Gravity Separators								
18. Centrifugal Separators								
19. Product Pumps and Valves	8.5×10^4	8.5×10^4	2.16×10^6	7.4×10^6	3.59×10^8	4.42×10^8	3.59×10^8	1.26×10^9
20. Filters			5.8×10^5	1.5×10^6			2.17×10^6	5.1×10^6
21. Flaker Drum, Belt Flaker							2.2×10^7	4.8×10^7
22. Distillations, Solvent Recovery			2.56×10^5	2.56×10^5			7.2×10^2	1.72×10^3
23. Mix-Melt Mix Operations	3.48×10^4	5.17×10^4	1.05×10^4	1.05×10^4	3.98×10^8	6.51×10^8	1.57×10^8	4.02×10^8
24. Packaging/Filling (Dry)	3.63×10^4	8.27×10^4	1.1×10^5	3.6×10^5	1.8×10^8	3.3×10^8	3.14×10^8	2.1×10^9
25. Machining	3.4×10^3	3.4×10^3	8.2×10^3	2.1×10^4	3.37×10^8	4.25×10^8	1.87×10^7	5.5×10^7
26. Wet Scrubber			2.7×10^4	4.8×10^4			5.6×10^8	6.26×10^8
27. All Operations	3.81×10^4	7.26×10^4	6.81×10^5	2.67×10^6	2.48×10^8	4.23×10^8	2.6×10^8	1.82×10^9

Key: $\bar{x}$ = mean value
$\bar{x} + \sigma$ = mean plus one standard deviation

Table 6

Comparison of historical accident data with engineering analysis (concl)

Process operation	ESD (joules)				Local thermal (°C)		Regional thermal (°C)				Impingement (m/sec)		Intermediate scale impact (m/sec)	
	Historical		Eng. anal.		Eng. anal		Historical		Eng. anal.		Eng. anal.		Eng. anal.	
	$\bar{x}$	$\bar{x}+\sigma$	$\bar{x}$	$\bar{x}+\sigma$	$\bar{x}$	$\bar{x}+\sigma$	$\bar{x}$	$\bar{x}+\sigma$	$\bar{x}$	$\bar{x}+\sigma$	$\bar{x}$	$\bar{x}+\sigma$	$\bar{x}$	$\bar{x}+\sigma$
1. Belt Conveyors			0.03	0.03					371	371			5.49	5.49
2. Bucket Conveyors														
3. Screw Conveyors														
4. Pneumatic Systems											14.8	2.3		
5. Hoppers	1.26	1.26	3.14	11.4							2.14	5.17		
6. Tote Bins			0.108	0.405									5	5
7. Screening	0.0063	0.013	0.0025	0.006	177	177					7.8	12.8		
8. Pressing														
9. Extrusion, Rolling							167	167						
10. Mills	0.014	0.014												
11. Glazing, Coating, Batch Drum			0.022	0.045					248	248				
12. Dryer	0.0084	0.0163	0.0051	0.0096			384	481	125	134				
13. Melt Pour, Casting	0.5	0.5					327	423			0.112	0.13		
14. Chutes														
15. Reactors							319	415						
16. Wash, Mix. and Hold Tanks							465	465	60	60	3.72	3.72		
17. Gravity Separators							222	222						
18. Centrifugal Separators							222	222						
19. Product Pumps and Valves					748	1071	250	250	158	277	20	26		
20. Filters									154	200	4.88	4.88		
21. Flaker Drum, Belt Flaker														
22. Distillations, Solvent Recovery														
23. Mix-Melt Mix Operations	0.014	0.014							115	124				
24. Packaging/Filling (Dry)	0.107	0.45	0.0116	0.017					180	180			4.3	4.3
25. Machining	0.26	0.26	0.29	0.29					60	60				
26. Wet Scrubber			2.4×10^{-5}	2.4×10^{-5}							80.5	80.5		
27. All Operations	1.06	4.37	0.742	4.71	748	1071	328	432	146	230	21.3	42.8		

Table 7

Inprocess potential energies

Process operation / Stimulus type	Local impact (j/m^2)	Impingement (m/s)	Rubbing friction (w/m^2)	ESD ignition (j)	Regional thermal (°C)	Local thermal (°C)
1. Belt conveyors	5.3×10^4	10	4.9×10^8	0.03	183	1000
2. Bucket conveyors	5.3×10^4	10	4.9×10^8	0.017	183	1000
3. Screw conveyors	5.3×10^6	10	4.9×10^7	0.017	100	200 C
4. Pneumatic systems	2.0×10^4	23[a]	1.1×10^8	1.0	100	NA
5. Hoppers	5.3×10^4	10	4.9×10^8	12.0	100	1000
6. Tote bins	5.3×10^4	10	4.9×10^8	12.0	100	1000
7. Screening	5.3×10^4	10	4.9×10^8	1.0	100	177
8. Pressing	6.76×10^4	5	4.9×10^8	0.017	100[a]	1000
9. Extrusion, rolling	6.76×10^4	NA	4.9×10^8	0.017	340[a]	1000
10. Mills	5.3×10^4	10	4.9×10^8	NA	183	200
11. Glazing, coating, batch drum	5.3×10^4	10	4.9×10^8	0.045	100[a]	1000
12. Dryer	5.3×10^6	10	4.9×10^8	0.017	431[a]	1000
13. Melt-pour, casting	4.4×10^4	NA[a]	4.9×10^8	0.017	423[a]	1000
14. Chutes	5.3×10^6	10[a]	4.9×10^9	0.017	100	1000
15. Reactors	4.4×10^6	NA	5.34×10^9	0.017	Special	NA
16. Wash, mix and hold tanks	4.4×10^6	NA	2.54×10^8	0.017	a	NA
17. Gravity separators	5.3×10^4	NA	4.9×10^8	0.017	a	NA
18. Centrifugal separators	5.3×10^6	NA	4.9×10^9	0.017	a	NA
19. Product pumps and valves	1.0×10^6	NA[a]	1.0×10^9	0.017	a	NA
20. Filters	7.4×10^6	26[a]	1.26×10^8	NA	227[a]	1071
21. Flaker drum, belt flaker	5.3×10^4	NA	4.9×10^8	0.017	200[a]	NA
22. Distillations, solvent recovery	5.3×10^5	10	4.9×10^3	0.017	183[a]	1000
23. Mix-melt mix operations	2.6×10^4	NA	1.72×10^8	0.017	a	NA
24. Packaging/filling (dry)	5.3×10^5	NA	6.5×10^9	0.017	124[a]	1000
25. Machining	3.6×10^4	10	2.1×10^8	1.0	183	1000
26. Wet scrubber	2.1×10^4	NA[a]	4.25×10^8	1.0	183	1000
	4.8×10^4	60[a]	6.26×10^8	0.017	a	NA

[a]Use operating conditions + 20 percent

that this list should be carefully scrutinized. It is based on somewhat weak data. For example, the statistical analysis of historical data only roughly sets a lower bound for the inprocess stimulus energies and many of the hazards analysis values might be considered "back of the envelope" estimates. Many process operation--stimulus type categories had no entries and a rough estimate or extrapolation from another category had to be made. As will be seen later, these values are quite important in the sensitivity portion of the classification procedure. The sensitivity class is assigned based on a safety factor, SF, defined

$$SF = \frac{\text{Sensitivity Test Energy}}{\text{Inprocess Energy}}$$

The denominator is obtained from Table 7, so a misleading entry in Table 7 can wrongly classify the sensitivity of the material. Fortunately, the major classification (NATO-UN type) is based on the effects testing, not sensitivity testing. The sensitivity evaluation is merely used as an indicator of the urgency of providing the safety features defined by the effects evaluation.

Not all stimuli are expected to be a problem for each of the process operations listed in Table 7. In the final procedure, a table is provided showing which sensitivity tests must be done for materials in each process operation. The tests required for each process operation were chosen by asking, "what initiation stimuli are credible and must be considered for this operation?" A table was developed in this way with the background gained from the historical data and hazards analysis reports. The table is presented in Appendix C. This was used to select those sensitivity tests which make sense for each of the process operations considered.

SURVEY OF EXISTING TESTS

To determine what test methods are already available and possibly useful in classifying inprocess materials, a survey of past and existing tests was conducted. A tremendous variety of test methods exists. Each laboratory has its own special purpose tests and versions of the more standard tests. This survey of test methods was certainly not all inclusive, but should be representative of tests with potential usefulness to this hazards classification application. The reports listed in Table 8 were surveyed. The tests which were uncovered will be summarized for each general type of test. Appendix B (extracted from Reference 2) provides descriptions of many of the tests discussed in this section.

Small Scale Impact

This category of tests must evaluate the likelihood of initiation of the sample material to a localized impact stimulus such as from a dropped tool, a person hammering, a dropped cover, an agitator impact, a part failure during operation, a person chipping off residue, etc. A variety of drop weight impact mechines exists. These include versions developed at the Bureau of Mines (BuMines), Naval Ordnance Laboratory (NOL), Los Alamos (LASL), Naval Weapons Center (previously NOTS), Picatinny Arsenal (PA), Bureau of Explosives (BuE) and Lawrence Radiation Laboratory (LRL). Variations in the machines include:

- unconfined sample versus sample in a cup
- smooth surface versus surface with grit
- direct impact versus impact through a striker/plunger
- matched drop weight to striker weight versus small striker
- different materials of construction
- various methods of preparing the sample
- results in terms of drop height versus other recording techniques.

Other localized impact tests include a small scale "flying" plate impact test (e.g., Table 8, Source 8-23), the bullet impact test (Table 8, Source 8-2), the LASL large scale (SPIGOT) impact test (Table 8, Source 8-3) and the thin film propagation test.

When deciding which local impact test has most promise for hazards classification of inprocess materials, several requirements of the test were set. First, the apparatus must be capable of applying an impact stimulus which is a reasonable full scale simulation of actual localized impact situations. This means that the test impact area should be on the order of the impact areas produced by the cases listed above (dropped tool, etc.) and the apparatus' maximum impact energy per unit area

Table 8

Reports surveyed for hazards classification tests

8-1 Schwartz, A. C., "Flyer Plate Performance and the Initiation of Insensitive Explosives by Flyer Plate Impact," SAND 75-0461, Sandia Laboratories, December 1975.

8-2 "Safety Performance Tests for Qualification of Explosives," NAVORD OD 44811, Volume 1, Naval Ordnance Systems Command, 1 January 1972.

8-3 Walker, G. R. (ed), "The Technical Cooperation Program, Manual of Sensitiveness Tests," Canadian Armament Research and Development Establishment, February 1966.

8-4 Dorough, G. D., et al., "The SUSAN Test for Evaluating the Impact Safety of Explosive Materials," UCRL 7394, University of California, Livermore, August 1965.

8-5 King, P. V. and A. H. Lasseigne, "Hazard Classification of Explosives for Transportation, Evaluation of Test Methods - Phase I," Department of Transportation, Final Report TSA-20-72-5.

8-6 Lasseigne, A. H., "Hazard Classification of Explosives for Transportation, Evaluation of Test Methods, Phase II," Department of Transportation, Final Report TES-20-73-2, May 1973.

8-7 Wilcox, W. R., "Evaluation of Test Methods for Pyrotechnic Hazard Classification," NASA National Space Technology Laboratories, Contract No. NAS8-27750, Edgewood Arsenal Contractor Report EM-CR-74051 (EA-4001), March 1975.

8-8 Cook, M. A. and R. T. Keyes, "Large Scale Drop and Projectile Impact Sensitivity Tests of Nitromethane," Report No. 11-NM2, Intermountain Research and Engineering Co., Inc., Fuly 1958.

8-9 Cabbage, W. A. and T. W. Erving, "A Compilation of Hazards Test Data for Propellants and Related Materials," RAD 100.10, Final Engineering Report on Production Engineering Project PE-489 (Preliminary), AMCMS Code 4932.05.4289.

8-10 Avramic, L., et al. (ed), "Proceedings of the Conference on the Standardization of Safety and Performance Tests for Energetic Materials - Volume I," ARRADCOM Special Publication, ARLCD-SP-77004, September 1977.

8-11 Baker, W. E., "A Review of Current Hazards Classification Test Methods," in Proceedings of the Conference on the Standardization of Safety and Performance Tests for Energetic Materials.

8-12 Dinsdale, V. T., "Hazard Evaluation of Solid Propellant Systems from Research to Missile Flight", paper presented at the American Ordnance Association Meeting held at Wright-Patterson Air Force Base, Ohio, 22-24 September 1964.

Table 8

Reports surveyed for hazards classification tests (concl)

8-13 Domalski, E. S., "Test Methods for Assessing the Thermal Instability of Hazardous Materials," in Proceedings of the Conference on the Standardization of Safety and Performance Tests for Energetic Materials.

8-14 Leining, R. B., et al, "Air Launched Missile Motor Behavior," AFRPL-TR-78-54, Technical Report (Special) prepared for Air Force Rocket Propulsion Laboratory, Director of Science and Technology, Air Force Systems Command, Edwards AFB, California, August 1978.

8-15 Mason, C. M., et al., "Drop Weight Testing of Explosive Liquids," Report of Investigation 6799, U.S. Department of the Interior, Bureau of Mines, 1966.

8-16 Napadensky, H. S. and R. Joyce, "Development of Hazards Classification Data on Propellants and Explosives," AARADCOM Contractors Report ARLCD-CR-78035, November 1978.

8-17 Nestle, W. R., "Formulation of Hazard Evaluation Indices for Pyrotechnic Processes," NASA National Space Technology Laboratories, Contract No. NAS8-27750, Edgewood Arsenal Contractor Report, EM-CR-74052 (EA-4D11), March 1975.

8-18 Pollack, M. E. and R. L. Wagner, "Development of an Impact Sensitivity Test for Cast and Pressed Explosives," Technical Report No. 2209, Ordnance Project No. TA3-5002A, Department of Army Project No. 5A04-01-011, Picatinny Arsenal, Dover, NJ, June 1956.

8-19 "Proceedings of the International Conference on Sensitivity and Hazards of Explosives," Explosives Research and Development Establishment, Waltham Abbey, Essex England, 1963.

8-20 "Some Unsolved Problems of Explosive Sensitivity," UCRL-7898, University of California, Livermore, June 1964.

8-21 Sumner, J. F., "A Rotary Friction Sensitiveness Test for Explosives," in Proceedings of the Conference on the Standardization of Safety and Performance Tests for Energetic Materials.

8-22 "Technical Bulletin TB 700-2, Explosive Hazard Classification Procedures," Department of the Army, 19 May 1967.

8-23 DeMella, D. et al, "Sensitivity of Cased Charges of Molten and Solid Composition B to Impact by Primary Steel Fragments," Technical Report 4975, Picatinny Arsenal, Dover, New Jersey, June 1976.

should be at least as high as the maximum level listed in Table 7. Second, the sample should be in the same physical form as it exists in the actual process. It should not be modified for the test. Third, impact should be against a rigid anvil so that the stimulus can be clearly defined. Other considerations such as the existence of grit could also be important but have not been addressed in this study. These criteria can be met fairly easily by modifying almost any of the drop weight machines or by using a modified version of the flying plate impact test. IITRI has three types of drop weight machines. A machine based on the Bureau of Mines design was determined to be able to produce the impact energies per unit area listed in Table 7. It was decided to modify this machine for the local impact testing. Reference 4 presents an experimental evaluation of the effect of varying the ratio of the drop weight to the intermediate weight for rigid samples (essentially impact into a rigid surface). It was concluded in Reference 4 that the most effective transfer of energy to the sample is achieved with matched (equal mass) drop weight and intermediate weight. Since most inprocess materials (unmodified) are "soft" samples, we conducted an experimental evaluation to determine if the same was true for "soft" samples. As will be presented later, it was found that for "soft" samples a better transfer of energy will occur if the intermediate weight is small compared to the drop weight. Besides modifying or removing the intermediate weight, the thrust of our experimental evaluation of the local impact test concentrated on developing one or more sample holders which most realistically represent local impact onto sample materials in the form that they exist in the actual process. In this sense, the local impact test becomes a simulation of the real impact scenario rather than a comparison of the chemical composition's impact sensitivity when the samples are prepared (generally modified) to all have the same physical form.

Impingement

The impingement test is to evaluate the material's sensitivity to particle-particle and particle-wall impacts in pneumatic conveying systems, cyclone separations, jet mills, etc. It also considers ignition sensitivity of particles falling from one process vessel into another. Free fall and propelled impingement tests are described in Source 8-1. In the propelled impingement test, the sample is injected into a moving air stream. The air carries the sample at some measured velocity onto a target plate. In the free fall test, the sample is dropped from a known height onto a horizontal or angled target plate. In both cases, light flashes and noise are used to indicate positive reactions. These tests are judged to be realistic simulations of the actual inprocess stimulus and are suitable as they presently exist for hazards classification of inprocess materials.

Container Penetration Tests

These tests simulate the penetration of a container by a rod like protrusion, for example a fork lift penetrating a process vessel. The

NOTS and NWL large scale impact tests (Table 8, Source 8-3) both involve a line of three rod protrusions attached to a drop weight dropped onto a cylindrical container laying on its side. It is felt that the likely ignition mode in these tests is the production of hot metal fragments. The production of such hot metal fragments is probably adequately represented by a thermal test. The container penetration tests realistically simulates only one type of accident scenario (vessel penetration) and will not be considered further in this study.

Regional Impact

This type of test is to evaluate the sensitivity of a material to impact over an area (essentially one-dimensional impact). This is representative of a container filled with energetic material dropped on its side, or overdesign operation of a hammer mill breaking up large chunks of material. A pass-fail form of this type of test is the 40 foot drop test (Table 8, Source 8-3). The bottle drop test or a more generalized version (container drop test) is a similar type of test. The large scale flyer plate (Table 8, Source 8-1) and the SUSAN tests (Table 8, Sources 8-2 and 8-4) can be used to evaluate this stimulus type in a more controlled manner. In our initial evaluation, the flyer plate test was selected as most appropriate for hazards classification. Since SUSAN test results can be correlated quite well to flyer plate results, the SUSAN test would also be acceptable. However, when looking at Table 7, under the Flyer Plate heading, it is seen that the inprocess impact velocities are quite low (10 and 20 m/s). It is quite unlikely that any samples would be initiated at such low velocities, and regional impact was eliminated from the list of credible ignition stimuli.

Sensitivity to Shock Wave

Several tests are designed to evaluate a materials sensitivity to pressure wave initiation. The different gap tests (e.g., card gap) are well suited for this purpose. The wedge test (Table 8, Source 8-2) also provides a good technique for evaluating the sensitivity of a material by exposure to a shock wave. Flyer plate and SUSAN tests can also be used to evaluate ignition by a shock wave.

In developing the hazard classification procedure it became clear that all possible hazard scenarios could not be addressed without making the procedure too complex to be practical. Limitations of the procedure's applicability had to be defined. Concerning the sensitivity evaluation, two general categories of ignition modes exist. The first category includes ignitions which originate in the process material being evaluated, i.e., not due to an initiation which originates elsewhere such as in an adjacent process vessel. Ignitions originating in the process vessel being evaluated are considered "primary" ignition modes. The second category of ignition modes, those originating elsewhere, are denoted "secondary" ignition modes. The process material where the ignition originated is the one that is responsible for

initiation, not the process material being evaluated. For this reason, secondary ignition modes such as by shock wave, by fragment impact, by fire brands, by massive flame impingement, etc., were excluded from the sensitivity evaluation.

Small Fragment Impact

A number of tests have been designed which characterize ignition by small fragments or bullet type projectiles. These include the small scale flying plate impact test (e.g., Ref. 6) and the bullet impact test (Table 8, Source 8-2). As with the shock sensitivity tests, the small fragment impact tests characterize a secondary initiation mode and will not be considered further.

Rubbing Friction Tests

Many types of tests have been used to evaluate the friction initiation sensitivity of energetic materials. In the literature reviewed for this program, five general types of friction tests could be identified. These are (1) the sliding strip and sliding block tests, (2) the pendulum tests, (3) the bowl type rotary friction tests, (4) the "pony brake" type rotary friction tests and (5) the bulk material friction tests. These tests are illustrated in Figure 2, with positive and negative aspects noted. While selecting the most appropriate friction test for this hazard classification procedure, the following requirements of such a test were considered to be of prime importance. First, the test must be able to accomodate a wide variety of material forms (i.e, powders, liquids, slurries, pastes, strands, etc.). For "fluid" material forms such as powders, liquids, slurries and pastes, the test should simulate two materials of construction rubbing across each other in the presence of the sample. For samples which consist of fairly large individual pieces such as pellets or strands, the sample should be rubbed across a material of construction's surface. Second, since real friction loads can be either long or short duration, the test should be able to apply the frictional load for both long and short durations. Third, (and most important) the frictional load must be well characterized and quantifiable. We assume that frictional ignition is related to a heating process, either very localized or over the contact area. In either case, the most pertinent parameter for correlating test data is the power (energy per time) dissipated per unit contact area. The duration of the frictional loading is also an important parameter. These parameters must be measurable in the test. Tests which provide the data which characterize the power per unit area and duration in a clean way are clearly the most desirable tests. In addition to the above factors, it is also desirable that the test be simple, inexpensive and easily operated.

For the devices shown in Figure 2, the following conclusions can be reached. The sliding strip and sliding block tests are simple and exist at many organizations. These are important advantages. The major

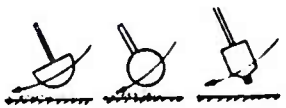
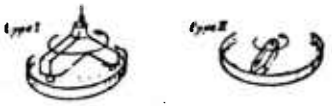
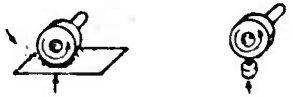
Type of Friction Machine	Illustration	Good Aspects	Bad Aspects
Sliding strip (drop weight drive) and sliding block (pendulum drive)		1. Many organizations (including IITRI) have these 2. Could be easily modified to test whole pellets, strands, etc 3. Can get high pressures using wheel and doesn't need high pressures using pellets, strands, etc. 4. Adaptable to all material forms 5. Can measure normal force and estimate area	1. Difficult to measure velocity-time function 2. Short run time. Need expensive modifications to increase friction duration 3. Leaves powder or liquid behind (single pass) 4. Thin liquid layer is not realistic and thick layer is difficult to contain.
Pendulum, with shoe or wheel		1. Simple apparatus - like modified Charpy tester 2 Adaptable to all material forms	1. Very short duration 2. Single pass 3. Can't measure normal force, velocity, or contact area, i.e., no parameter values are known
Rotary, bowl type		1. Adaptable to all material forms 2. Can handle long and short durations 3. Can measure friction force, velocity, contact area and contact duration 4. Multiple passes are possible	1. Due to balance, must always have two contact points (not as easy to interpret as one contact) 2. Type II has limited normal force 3. Costly due to size and balance 4. Possible splashing and dusting problems 5. Can't handle extremely short durations
Rotary, pony brake type		1. Simplified basic design (essentially a pony brake) 2. Adaptable to all material forms 3. Can measure friction force, velocity, contact area and duration	1 A possibly complex design would be required to handle dust or spray produced (e.g., (a) continuous feed and vacuum or (b) recycling of sample around wheel). 2. Can't handle extremely short durations
Bulk material tests		Not applicable for hazards classification of in-process materials	Not applicable for hazards classification of in-process materials

Fig 2 Comparison of friction sensitivity tests

disadvantage is that the velocity-time function and frictional load are not provided by the test. In addition, the test as it exists at most facilities is only capable of providing very short duration loads. Because of its simplicity and availability, this type of test was evaluated in the previous project (Ref. 2). It was found difficult to quantitatively characterize the friction stimulus and for many material forms the material had to be altered to accomplish the test. For these reasons, the test was found to be undesirable. The pendulum tests, again, are simple but they do not provide the data required to quantify the frictional loads. The sliding strip, sliding block and pendulum tests are more suitable to ranking materials relative to some standard rather than finding the power per unit area-time relation for ignition. The bulk material test is a type of pendulum test for bulk materials and not applicable to testing most inprocess material forms. The two rotary friction test concepts are somewhat more complex and costly to run but are the only tests which cleanly quantify the required parameters (contact duration and power per unit area in terms of relative velocity or r.p.m.'s, and frictional force or torque). The ability of the rotary tests to cleanly provide the required test data, where the other tests do not, is of major importance. The choice between the two rotary concepts is based entirely on which design can be adapted most easily. Problems in feeding and collecting dust from powder like samples in the "pony brake" configuration are considered to be much greater than problems associated with the bowl arrangement. Therefore the bowl arrangement was selected as the best choice for hazards classification. Thiokol has done work with both rotary machines (Ref. 7) and the design of the bowl type rotary machine for this program is based heavily on the Thiokol apparatus.

Electrostatic Discharge Evaluation

The electrostatic discharge evaluation has two parts. The first part of the evaluation characterizes the ability of the inprocess material to develop electrostatic charge. This is primarily related to the development of discharges within the sample material itself. The second part of the evaluation concerns the initiation sensitivity of the material to an electrical discharge. The basic techniques for determining charging susceptibility have been fairly well developed (Ref. 8). The technique involves applying an oscillating voltage across the sample and measuring permittivity and electrical conductivity. The ratio of permittivity to conductivity is the characteristic charge relaxation time and is indicative of the material's charging susceptibility. The only extension of existing methods which was required under the current program was ro measure these electrical properties for inhomogeneous materials such as pellets and strands using a large sample holder. Ignition by an electrical discharge in a layer of material is tested by passing well defined electrical discharges through a layer of the sample. Vapor or dust cloud ignition is evaluated using a Hartmann or Bartneckt type apparatus.

Localized Thermal Ignition Tests

Several tests consider the sensitivity to initiation by an intense localized thermal ignition source such as an incendiary spark produced by friction, from impact of a foreign material in a mechanical process operation, from welding, from a cigarette, etc. The hot wire igniteability test (Table 8, Source 8-2) was one viable option for characterizing this stimulus type. A second option is to mechanically produce sparks using a friction wheel like a grinder wheel. This technique exposes the samples to numerous random hot sparks. It is quite difficult to quantify this stimulus since many sparks are hitting the sample and some locations may be hit several times. Another technique is to heat well defined tiny metal balls in an electrical heater and drop the balls at a specified temperature ontc the sample material. This technique was determined to be most promising and was selected for experimental evaluation in this program.

Regional Thermal Ignition Tests

As the title implies, regional thermal tests evaluate the potential for initiation of a self sustained propagating exothermic reaction when the material is heated over a substantial volume. In a process plant, the material can be exposed to an elevated temperature in many ways. A system malfunction or operation error could result in the material in a heat exchanger type process vessel to be raised to above the design temperature. This will happen if there is a loss of cooling, loss of agitation, etc. Material could be spilled or sprayed and exposed to a hot pump or motor.

Some of the tests which have been used to evaluate this stimulus will be outlined. In the existing document TB 700-2, the copper block test and the self heating test are described. In the copper block test a 0.1 gram solid sample is heated at a controlled rate until ignited. The self heating test utilizes differential thermal analysis and "cook-off tests" to generate the constants in an equation which estimates the maximum temperature to which a cylinder of a given diameter can be exposed before a runaway chemical reaction will occur. Another cook-off test determines the lowest temperature at which a 5 milligram sample will "flash off" in 10 seconds. In the Wenograd test (also in TB 700-2), the sample fills a thin stainless steel tube. The tube is heated using a capacitor discharge and the samples "explosion temperature" is determined. In the Taliani test, also described in TB 700-2, the sample is placed in a fixed temperature heating block with pressure and rate of pressure rise monitored. The vaccuum thermal stability and chemical decomposition test holds the sample at 100°C in one version, 75°C in another, for at least 48 hours. Reactivity is indicated by gas evolution which must not exceed 2 milliliters per gram of sample during the 48 hours for acceptance. A test based on the same type of exposure (75°C for 48 hours) has been conducted by General Electric (Table 8, Sources 8-5 through 8-7). In that test, the sample was packed in a

tube, wrapped with heating tapes, capped at both ends and insulated. The elevated temperature exposure was produced using the heating tape.

For liquid samples, a variety of tests are described in ASTM standards. These include the ASTM D2155 autoignition test, the ASTM E136-65 noncombustibility test for elementary materials, and the ASTM D1929-68 test for ignition of plastics. These tests were all oriented toward materials which do not contain their own oxidizer and were not considered general enough. The most promising tests in terms of simplicity, generality and availability of equipment at many laboratories were the differential thermal analysis (DTA) and differential scanning calorimetry (DSC). The DSC was experimentally evaluated in the initial project and was found to be suitable for hazards classification of inprocess materials.

In addition to the tests outlined above, a number of thermal tests have been developed which expose the sample to an engulfing fire. These include the TB 700-2 external heating "Test C", and the GE ignition and unconfined burning test (Table 8, Sources 8-5 through 8-7). Since these tests assume a fully developed fire already exists the stimulus is a secondary ignition source and not in line with our philosophy of considering only primary ignitions for the sensitivity evaluation.

Critical Size Tests

The "critical diameter" test (Table 8, Source 8-2) exposes a sample confined in a tube to a flat pressure wave produced by detonating a condensed explosive at one end of the tube. This test addresses the question "can the sample propagate a detonation which has already been established?" The "critical depth" (layer thickness) test exposes a layer of sample material to a pressure wave at one end using an explosive booster to determine what thickness the layer must be in order to propagate an established detonation. In this report, this test will be referred to as the "critical layer thickness" test. The wedge test is a modification of the critical depth test in that the layer thickness decreases away from the explosive initiating charge. The point at which the reaction ceases gives a very conservative estimate of critical layer thickness in that the detonation travels a substantial distance before it dies out.

In the "critical length" or transitian test. The sample is packed in a tube and initiated at one end using a flame ignition source. The test is designed to determine the length required for a flame to transition into a detonation in a container of a given diameter. A version of this test designed to simulate material in a hopper type arrangement is called the critical depth or critical height test (Table 8, Source 8-9). In this report, we will refer to this type of test as the "tube transition" test. A parallel test can be accomplished for a layer of material. This test will be called the "layer transition" test in this report. All of the critical size tests were considered to be of potential value to the hazards classification procedure being developed and all of these tests, except the wedge test, were experimentally evaluated.

Mass Explosion Tests

To evaluate the effects produced by the detonation of the sample material, a "mass explosion test" is required. This type of test must be capable of evaluating the airblast (primarily) and fireball (secondarily) hazards imposed by the material. This type of test has been widely used and is commonly known as a TNT equivalency test. The sample is placed in the field with one or more string of pressure transducers positioned to measure the airblast pressure wave as it passes at various distances. The scaling laws for airblast are well established. To evaluate the fireball hazard, high speed movie coverage and slug calorimeters can be used.

The only question in conducting this type of test for hazard classification is concerned with the configuration in which the sample is placed. The most realistic configuration would be a scaled model process vessel. This generally is the technique used, because it provides the most realistic answer, and would always be acceptable for hazards classification if such a test were to be conducted anyway for another purpose. The problem with a scaled model is that properly scaling the actual process vessel can be somewhat complex and the actual process configuration may not be precisely known especially if hazard classification is done early in the development cyclc of a system. The alternatives are to use a standardized conservative cyclinder, sphere, or hemisphere. The hemisphere represents a fairly simple (one-dimensional) configuration to evaluate and was selected as most promising for hazards classification.

Fragment Evaluation

Fragments produced by the detonation of a material in a metal container are an important part of the overall mass explosion hazard. The fragment problem can be divided into three parts. First, the initial velocity must be determined. Next, the fragment size distribution must be known since the largest fragment has the potential (initial momentum) to go the farthest. Finally, the potential for damage of a fragment at a given size and velocity at a target must be characterized. To determine initial velocity, the Gurney constant (Ref. 9) can be experimentally determined using a cylinder expansion test (CYLEX) or a plate push test. To determine the fragment distribution, the fragments can be caught using sawdust, water, a fiber board barrier, an earth barrier, a layered cellotex-paper catcher, etc. The fragment ignition problem is being evaluated under ARRADCOM funding (Ref. 6). Since the airblast best characterizes the mass explosion hazard, a fragment evaluation was determined to be not necessary for hazard classification purposes.

Fire Effects

Several distinct fire hazards related to the process materials exist in process plants. These will be discussed in more detail later. With the mass explosion airblast and fragment hazards, a fireball producing a radiated thermal pulse provides a third aspect to the total

mass explosion hazard. If an open topped vessel containing process material is ignited, a fire plume may develop over the material. If the generation of hot gases is substantial enough (about the point at which the flame impinges on the ceiling), the total heating of combustibles in the enclosure will be sufficient to cause the nearly simultaneous ignition of all these materials. This point is generally called "flashover" in fire research. Even if flashover does not occur, the radiated heat flux from the flame may be adequate to ignite combustibles a distance across the room. Finally, if the material exists in the process in a layer, such as a conveyor or trough, fire spread along the layer to the process vessel at the other end is of concern. Many tests in the past have been done to evaluate the fire spread hazard, to assure that the detection-deluge system will respond quickly enough. These have been both small scale and nearly full scale simulations of the acual system.

Other Hazards

Other hazards not included in the discussion above also could be of concern in process plants. These include production of firebrands, toxicity and chemical compatibility. Although such other hazards may be important, it was decided to limit the scope of the procedure being developed in order to not overly complicate it. These additional hazards were considered to be beyond the scope of the procedure being developed.

TEST EVALUATIONS

The studies described in the previous three sections helped to mold the structure of the overall hazards classification procedure. In the procedure, the hazard was seen to consist of two parts: an evaluation of the materials sensitivity (how likely is an initiation to occur?) and an evaluation of the consequence of an initiation. Existing hazard classification procedures require sensitivity type testing but put the materials into a consequence related classification. That approach is not felt to be proper and was not followed here.

The sensitivity evaluation is to consist of quantitative sensitivity tests for stimuli which can exist in the different process operations. The test results (the materials sensitivities) are then to be compared to credible inprocess stimuli levels such as those listed in Table 7 in order to categorize the material's sensitivity hazard. Independently, an effects evaluation is to be completed in order to characterize the material's consequence severity. The effects evaluation classifies the material in categories such as are used in the NATO-UN system.

Based on the survey of test methods, using the historical DDESB accident report survey, a survey of hazards analysis reports, and a preliminary formulation of the overall procedure as it was envisioned early in the program, the following tests were selected for an experimental evaluation:

- Local Impact
- Rubbing Friction
- Local Thermal
- Regional Thermal
- Electrostatic Discharge
- Critical Diameter
- Critical Layer Thickness
- Tube Transition
- Layer Transition
- Mass Explosion
- Mass Fire
- Firespread

These tests were each evaluated using four sample materials. M1 strands were used to represent an extrusion process; M30 pellets represents a drying operation; RDX slurry corresponds to a conveying operation; and M26 paste was from a mixing operation.

The sample materials which were used were all from the previous program. By the time they were used on this project, they were undoubtedly quite different from their true inprocess form. For example, solvent concentrations were undoubtedly significantly lower than in the actual process. Therefore, no attempt was made to realistically classify the materials. Rather, the samples were used in order to try out the different test procedures to assure that the procedures are adaptable to the variety of material forms which can exist in process operations. In order to standardize the experimental evaluations, the specimen bulk densities* were fixed at the following values wherever practical throughout the testing:

M26 Paste Density = 0.829 gm/cm^3 (0.0299 lb/in^3)
M1 Strands Density = 0.45 gm/cm^3 (0.0162 lb/in^3)
M30 Pellets Density = 0.838 gm/cm^3 (0.0302 lb/in^3)
RDX Slurry Density = 1.114 gm/cm^3 (0.0347 lb/in^3)

NOTE: The RDX was used as received, rather than being mixed with water to obtain the true inprocess composition.

As will be seen in Section 6, the hazard classification procedure requires several additional tests not experimentally evaluated in this program. These tests include:

- Impingement ignition tests
- Flame ignition test
- Dust/vapor explosion test (Hartman or Bartneckt test)

These were judged to be fairly well established or simple enough not to require further evaluation under this project.

In the subsections which follow, each of the test methods which were experimentally evaluated will be discussed, including a description of the experiments conducted and the test results.

Local Impact Test

The local impact test is to determine the impact energy per unit area required to initiate the sample material. This stimulus corresponds to scenarios such as a dropped tool, a person hammering, a dropped cover, agitator impacts, part failure during operation, a person chipping off residue, etc.

* Bulk density is equal to the average mass per unit volume of the material as it is packed in a container, rather than the density within the individual grains, pellets, strands, etc.

The philosophy taken by IITRI on this, as well as all the other sensitivity tests, was to have the test be essentially a full scale simulation of the actual stimulus. The sample material should be in the same form that exists in the real process operation. The inprocess bulk density should be reproduced. The impact area should be representative of typical actual local impact situations. In the case of impact area, the results will depend somewhat on the area selected and a standardized impact area must be selected. In the impact test, as well as many of the other sensitivity tests, an attempt was made to have sufficient sample present, extending beyond the impact area, so that initiation and propagation of the reaction could be used as the criteria for a positive result. This is important in that we really do not care if a nonpropagating reaction occurs (e.g., only discoloration, gas evolution, slight burn, etc.). The reaction must be initiated and be able to propagate into the surrounding material for it to be significant.

IITRI has three types of impact machines which could be used for this evaluation. The machine with the highest potential drop height was selected so that the inprocess energies given in Table 7 (up to 4.4×10^6 j/m^2) could be achieved. In actuality, the maximum energy per unit area achieveable with the machine was ultimately slightly below 4.4×10^6 j/m^2 (it was 3.97×10^6 j/m^2), but the machine's potential was well above what was needed for any of the four sample materials being evaluated.

In the present project, the machine was modified in three respects. First, an optical velocity sensor was positioned near the bottom of the drop in order to assure the proper impact energy was used to correlate the data. This was done because significant energy losses during the drop were suspected.

Figure 3 shows the actual impact velocity compared to the measured velocity. It is clear that significant energy losses occur. The extreme losses are probably due to some poor design features inherent in the particular IITRI machine design, however, it is likely that any machine will have some losses. The different losses between machines could in part account for poor correlation of drop weight impact test results between different machines. It is suggested that velocity at impact be measured and kinetic energy (based on the actual velocity) per unit impact area be used to correlate the data.

The second machine modification was to remove the intermediate weight, which is a basic part of the Bureau of Mines design. Work at the Bureau of Mines (Ref. 10 and 11) showed that the best arrangement for transferring drop weight energy to the sample most efficiently in a single pulse with a minimum of oscillation, is to use an intermediate weight of equal mass as the drop weight. That work was done specifically for pressed samples, so that the impact was essentially onto a rigid surface. Since most inprocess materials are softer targets, it was decided to evaluate the effect of the intermediate weight on

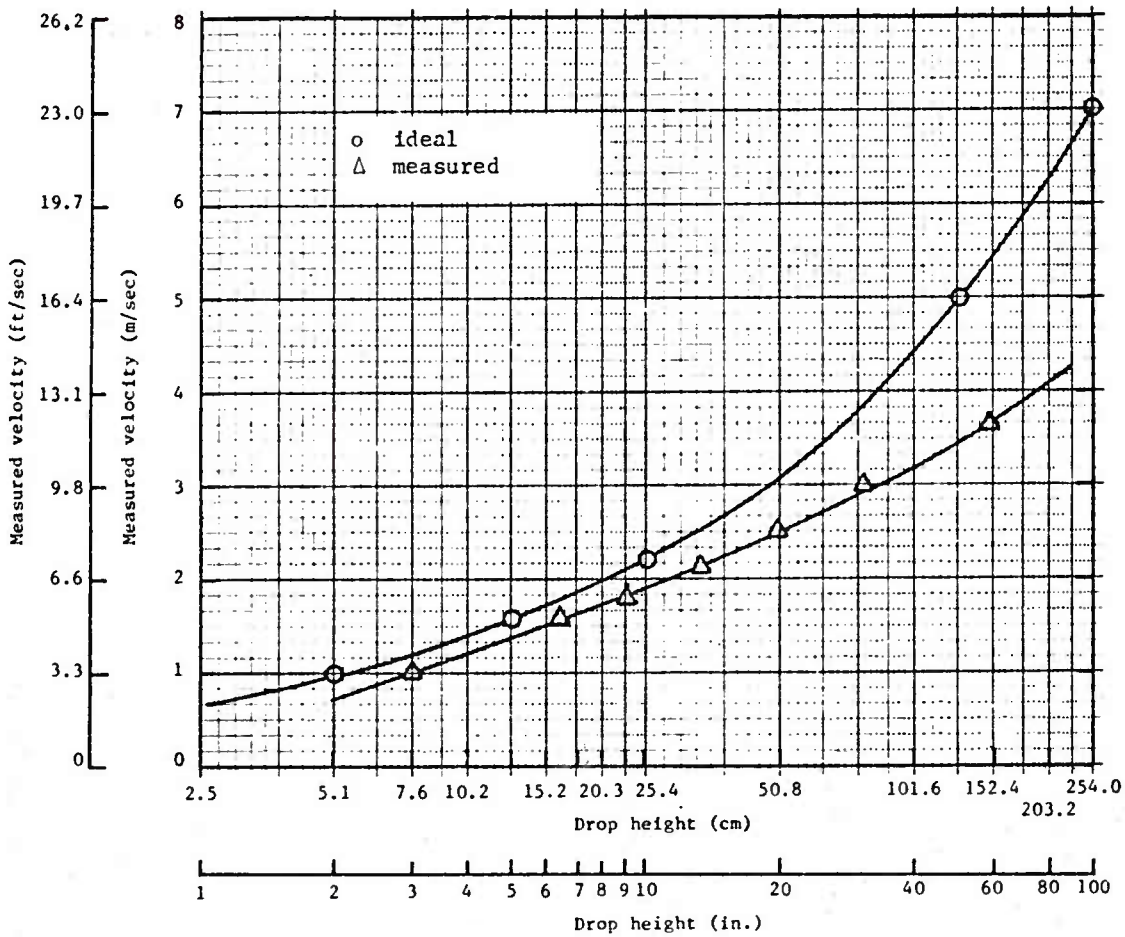


Fig 3 Comparison of ideal and measured impact velocity for IITRI drop weight impact machine

transferring energy to a series of soft targets. Neoprene, hard cardboard and lead were used. The ratio of intermediate weight to drop weight, R, was varied from 0.458 to 0.104. The results are presented in Figure 4.

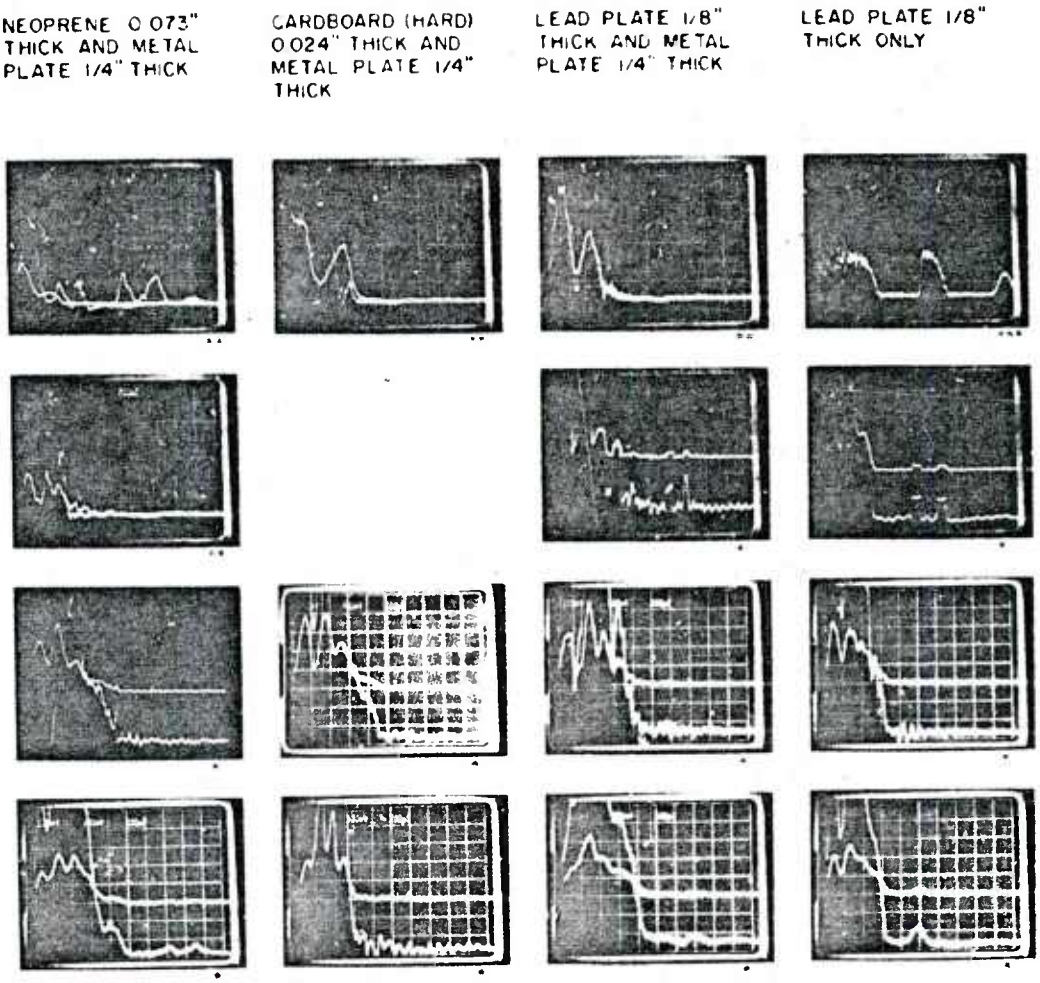
In the figure, force-time profiles (oscilloscope traces) are shown for a quartz force transducer mounted beneath the samples indicated. Going down each column, decreasing the ratio R, the force time profiles appear to be getting less oscillatory, forming a single pulse. Although the pulses are still not very clean, it appears that the best pulses are with the smallest intermediate weights. In addition, with a very small rigid intermediate weight (striker pin), the transfer of energy should be most efficient. The small striker pin arrangement also was most convenient for the sample holder designs which were evolving.

The third machine modification was to design one or more sample holder which could be used to simulate impact onto the inprocess materials as they exist in the actual process operations. The sample holders originally designed are shown in Figure 5. Type 1 was to be used for impact within a bulk of material. The material surrounding the impact location was there to show the ability of the reaction to propagate. It was found that the original type 1 holder was too large. A test with about 5 grams of the RDX sample resulted in a detonation totally destroying the sample holder and damaging the drop weight. In order to preserve the concept behind the type 1 holder (to be able to observe initiation as well as propagation) but use a smaller totally expendible holder, the design shown in Figure 6 was used.

The type 2 sample holder was found to be best for impact onto strands or pellets. The type 3 sample holder was designed after the adiabatic compression test of Reference 12. This sample holder is probably a good technique for testing liquid samples, particularly when comparing the sensitivity of liquids to the adiabatic compression stimulus of some reference material. In the context of the hazard classification procedure being developed, in which the test energy is to be compared to the inprocess energy, the meaning of the adiabatic compression test results could not be interpreted. It was decided to use the type 1 sample holder for liquid samples, since the stimulus could be put in terms of a well defined energy or power per unit area.

The Bruceton technique* was used for obtaining the 50 percent probability of initiation point for each sample material. The test results are summarized below:

* The test sequences generally included less than ten tests, therefore the estimated 50 percent point in each case is approximate.



* DUAL SWEEP, ONE AT 100mV/SQ, THE OTHER AT 500mV/SQ
** MORE THAN ONE TRIGGER (UTRI USED INTERNAL TRIGGERING)
*** QUESTIONABLE FORCE-TIME PROFILE (AREA OF IMPACT MUCH SMALLER THAN AREA OF PRESSURE TRANSDUCER)

Fig 4 Force-time traces of various ratios of intermediate mass to impact mass

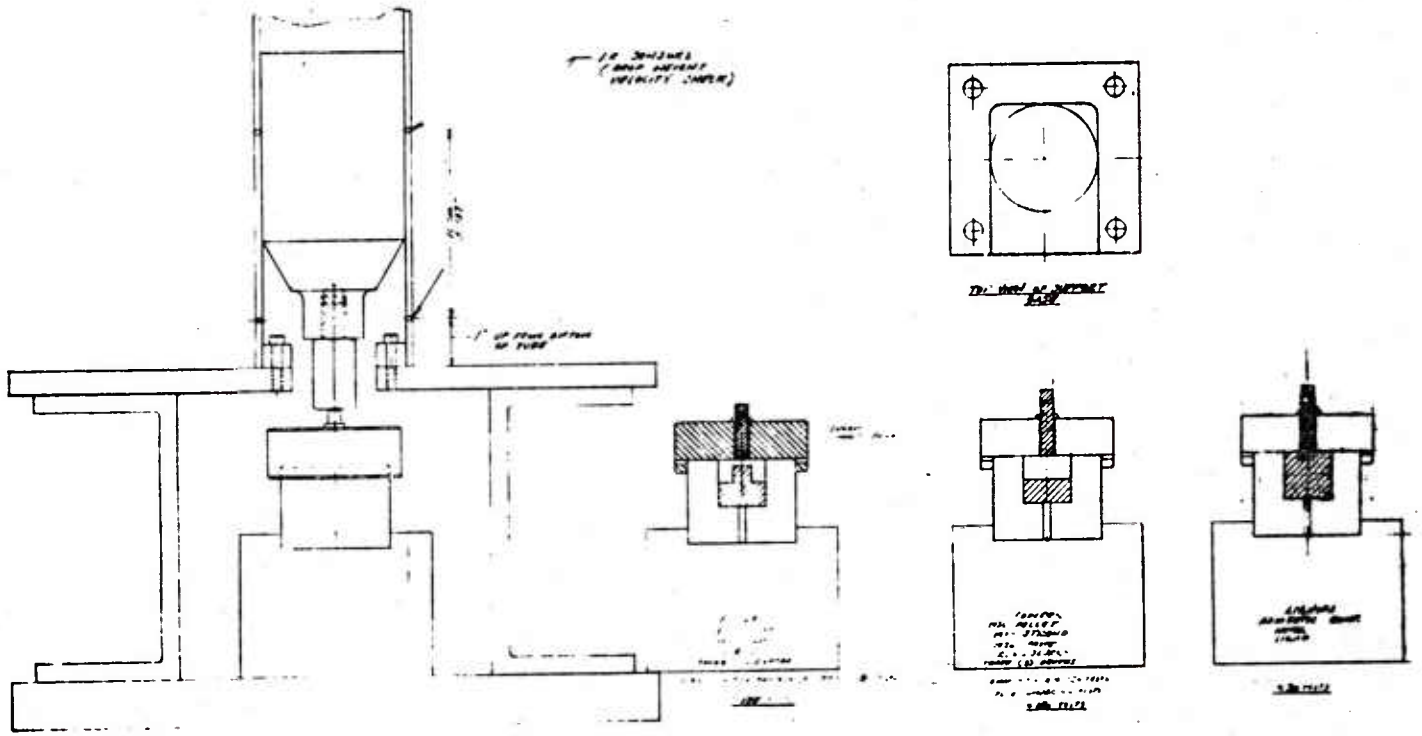


Fig 5 Modified impact tester and sample holders

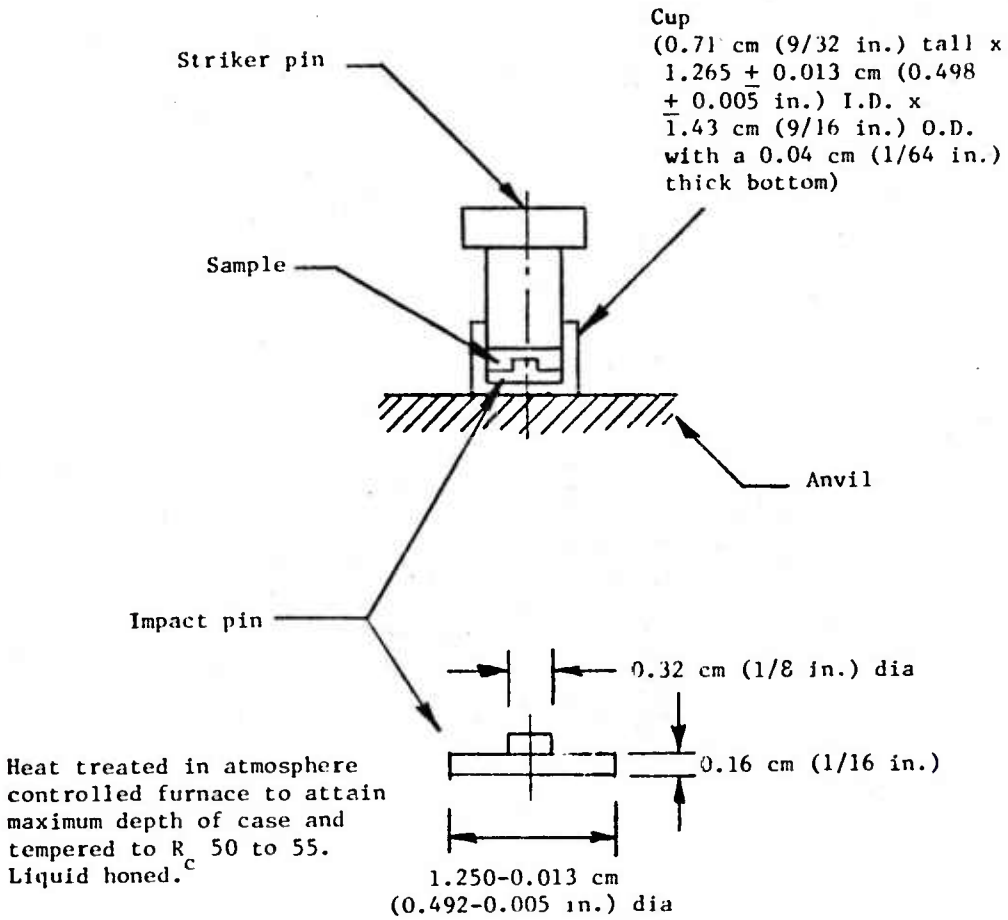


Fig 6 Modified type 1 sample holder

Sample Material	Inprocess Energy From Table 7 (j/m^2)	Sensitivity Test Energy (j/m^2)	Safety Factor
M1 Strands	6.76×10^4	$>3.14 \times 10^6$	>46
M30 Pellets	5.3×10^4	$>4.45 \times 10^5$	>8.5
M26 Paste	5.2×10^4	6.92×10^5	13
RDX Slurry	5.3×10^4	1.5×10^6	36
Nitromethane with 5% Ethylenediamine by Volume	-----	$>3.97 \times 10^6$	---

The Safety Factor is the ratio of the 50 percent probability energy per unit area obtained from the test to the inprocess energy (from Table 7). The safety factor is used to indicate whether or not the stimulus being evaluated (impact in this case) is expected to be a problem in the particular process operation. The "cutoff" valve for the Safety Factor is taken to be 3. For all the samples tested, the safety factor is well above 3 and impact is not expected to be a major problem.

Rubbing Friction Test

The rubbing friction test simulates initiation caused by two solid materials rubbing across each other. When the sample material being evaluated has fairly large individual pieces, such as chunks of material, pellets, or strands, the friction is between the sample material and an appropriate material of construction (e.g., steel representing the process vessel's wall). If the sample material is of a fairly fluid form, such as a fine powder, a slurry, a paste, or a liquid, the friction is between two materials of construction in the presence of the sample material. The sample may act as a lubricant in these cases. In either situation, initiation is expected to correlate adequately with energy deposition per unit time over a properly defined frictional contact area (i.e., power per unit area--watts per square meter). This translates into a rise in the temperature of the sample. If the temperature is high enough over a large enough volume of material, ignition and propagation will result.

As discussed in Section 4, there are many types of friction tests which have been used. In the previous program (Ref. 2), the strip friction technique was evaluated. This is illustrated in Figure 7. The strip friction apparatus has the advantage of simplicity and availability at many laboratories, however, it has two important disadvantages. First, the power dissipated per unit contact area is somewhat difficult to quantify in the test. The power dissipated varies with time and different histories are certain to influence the results. Second, in many cases, the sample must be significantly altered from its inprocess form in order to accomplish the test. This second problem is quite important and makes the strip friction test unacceptable for our purposes.

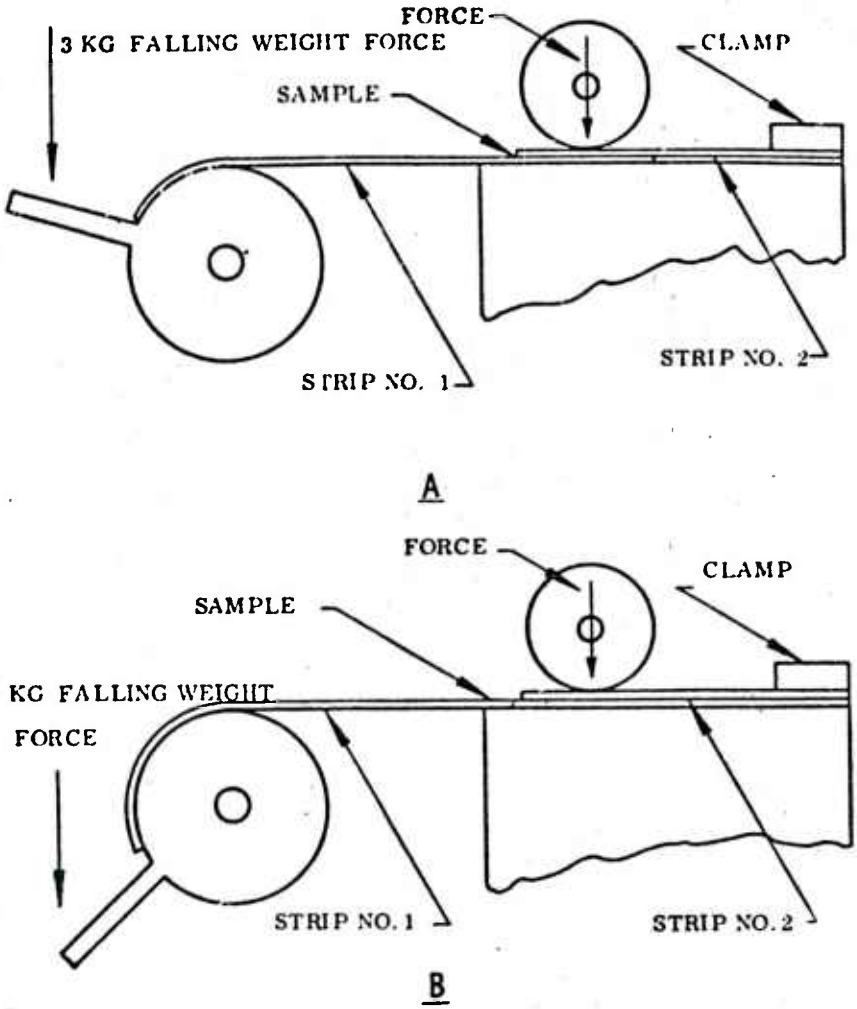


Fig 7 Strip friction test

After comparing the good and bad points of the different friction tests, the rotary friction concept based on one of Thiokol's designs (Ref. 7) was selected as the most promising. Thiokol's test arrangements are shown in Figure 8 and the apparatus used for this project is illustrated in Figure 9. The apparatus used for this project was merely to verify that the rubbing friction evaluation can be accomplished adequately using the rotary friction test concept. There is no question that the design used for this evaluation can be improved upon significantly.

As shown in Figure 9, a d.c. motor rotates the contact points over a steel plate. The contact points are two steel balls for powder or fluid samples, or two sample pieces for pellets, strands, etc. The steel plate represents the actual process material of construction and should be replaced by a realistic material of construction with a realistic surface finish if the process surface is not adequately represented by the steel plate. The steel plate is grooved as shown for tests on fluid samples. A torque sensor, which also measures revolutions per second, is fixed to the shaft between the pulley wheel driven by the d.c. motor and the contact points. Revolutions per second can be converted into tangential velocity by the formula

$$V = \pi D f$$

where D is the distance between contact points and f is the measured revolutions per second.

Torque is given directly from the sensor and can be converted into frictional force per contact point (2 contact points exist) by the formula:

$$F = \frac{T}{D} \qquad \text{where T is the measured torque.}$$

The power dissipated per contact point is then

$$P = FV = \pi f T$$

We are interested in power dissipated per unit area of contact. The contact area can be estimated from the following equation (Ref. 13):

$$A_c = \pi a^2 \qquad \text{where}$$

$$a = 0.721 \sqrt[3]{Pd\left(\frac{1-\nu_1^2}{E_1} + \frac{1-\nu_2^2}{E_2}\right)}$$

In the formula, P is the normal force applied to the ball. If the lever arm arrangement between the weight and the contact section is balanced and frictionless, P is merely the weight divided by two since there are two contact points. In the formula, ν_1 and ν_2 are Poisson's ratios for the balls and the contact surface respectively; E_1 and E_2 are the moduli of elasticity for the balls and surface respectively.

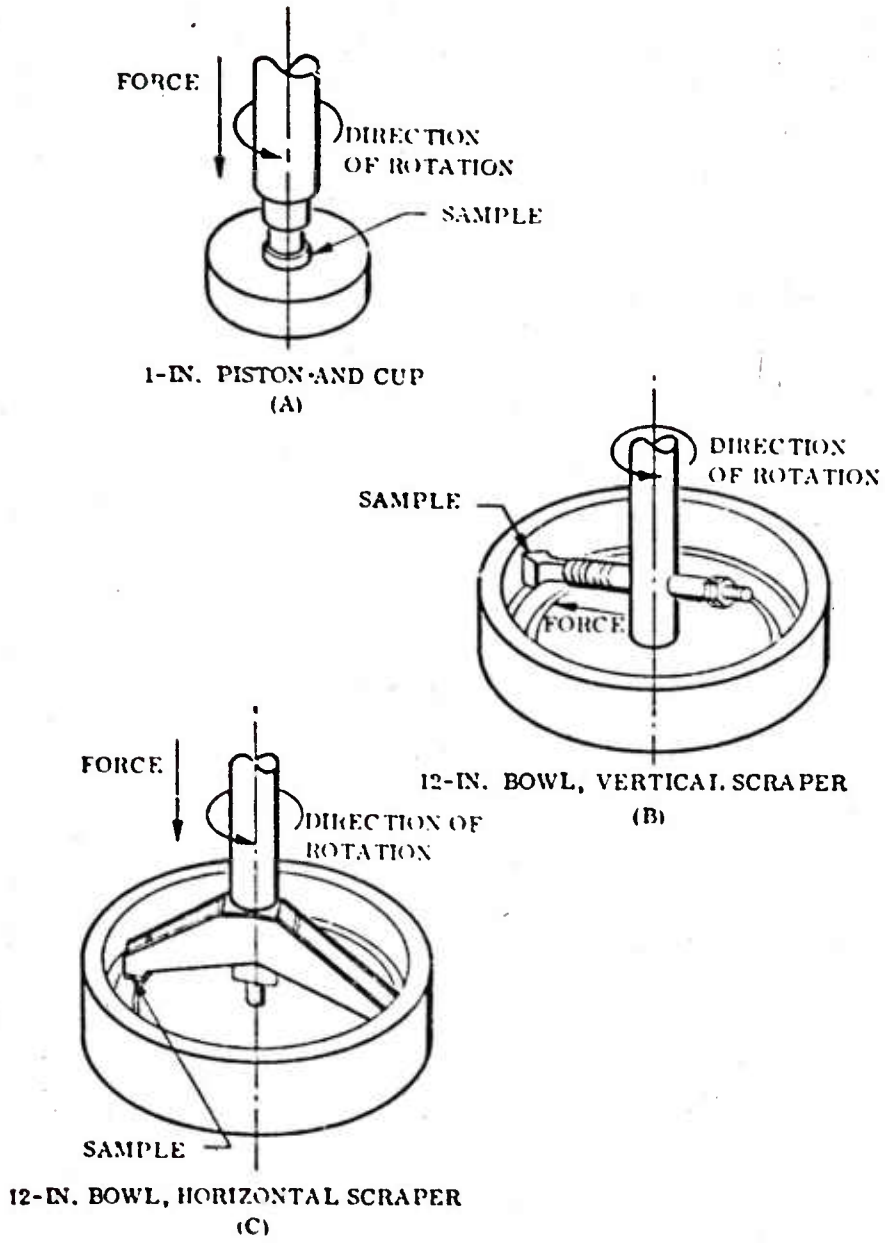


Fig 8 Thiokol's rotary friction test
(from Ref 7)

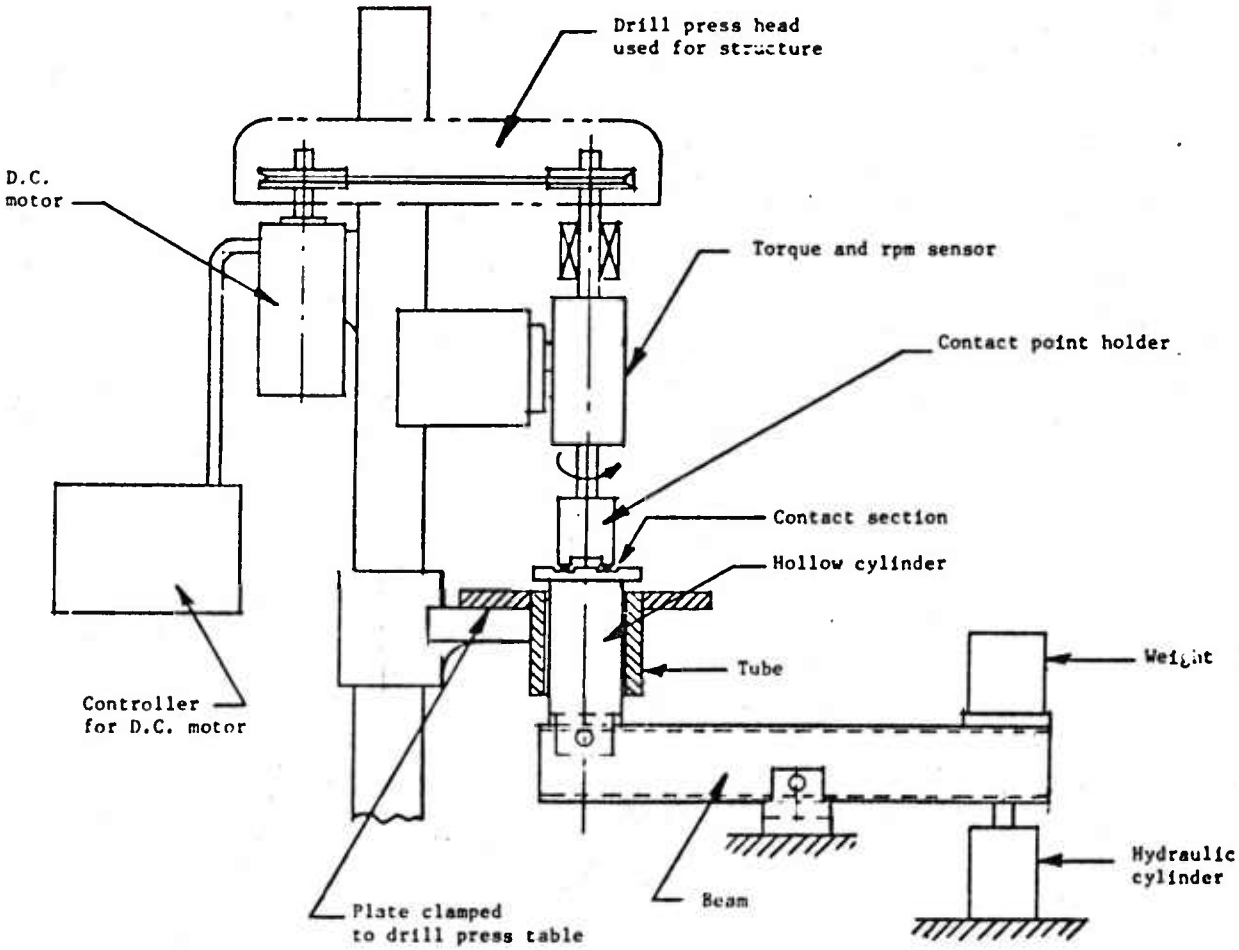


Fig 9 IITRI rotary friction apparatus

Combining the above equations gives an expression for frictional power per unit contact area:

$$\frac{P}{A_c} = \frac{fT}{a^2} \quad \text{with}$$

a = sample contact radius, for in homogeneous samples such as pellets

$$a = 0.721 \sqrt[3]{Pd\left(\frac{1-\nu_1^2}{E_1} + \frac{1-\nu_1^2}{E_2}\right)}$$

for homogeneous samples such as powders, slurries, pastes and liquids

Using the apparatus shown in Figure 9, tests were conducted on the four sample materials. The tests which were accomplished are listed in Table 9. Ignition was not observed in any case tested. Several significant observations were made during the tests. First, for pellets and strands, it was found that due to the large frictional contact area, power per unit area was always orders of magnitude below the inprocess values listed in Table 7. The sample merely failed structurally producing numerous "shavings". Based on this result, it appears that in the actual process a pellet or strand would first fail structurally and then the shavings produced would get into moving parts and be acted upon. Therefore, the samples were retested in the failed form using the steel balls and higher loads.

Second, it was found that the steel balls wear down very quickly during the test. In terms of frictional power per unit area, there is an initial very high load but the load drops to a much lower steady value because of the greatly increased frictional contact area. If this high initial peak dominates the initiation process, the sliding block may end up being just as realistic as the rotary concept, that is if the frictional force versus time relation could be quantified in the sliding block machines. The simpler device would naturally be the more desirable in that case and further work in this area to quantify the stimulus produced by the sliding block machine could be quite worthwhile.

Third, the steel balls and anvil surface deteriorated significantly during the tests. New balls were used for each test and the anvil's surface was refinished using number 200 emery paper after each test. Nevertheless, the anvil's surface was badly worn at the end of each set of tests and had to be remachined twice during the series. It is therefore suggested that the anvil be designed with a replaceable top plate to contact the steel balls. The top plate should be given a Rockwell C hardness of 60, and it should be replaced when significant wear becomes evident.

Table 9

Rubbing fraction tests conducted

Test	Sample material	Total normal force (kg)	Measured rpm	Measured torque (N-m)	Power per unit area (w/m^2)	Result
M30-1	M30 Pellets	18	211	1.11	$2.5x10^5$	Pellet deteriorates
M30-2	"	"	97	0.97	$1x10^5$	"
M30-3	"	"	321	0.97	$3.3x10^5$	"
M30-4	"	"	425(estimated)	-	-	"
M30-5	"	"	885	0.7	$6.6X10^5$	"
M30-6	"	29	189	-	-	"
M30-7	-	"	362	0.18	$6.9x10^4$	"
M30-8	-	-	571	-	-	"
M30-9	"	"	736	0.13	$1x10^5$	"
M30-10	-	"	971	0.14	$1.4x10^5$	"
M30-11	M30 shredded	35	1695	0.11	$1.15x10^8$	No reaction
M30-12	-	"	1639	0.11	$1.23x10^8$	"
M1-1	M1 Strands	18	160	0.2-0.26	$4.4x10^4$	Strands deteriorate
M1-2	"	"	361	0.15-0.33	$1.3x10^5$	"
M1-3	-	-	543	0.18	$1x10^5$	"
M1-4	"	"	740	0.19	$1.5x10^5$	"
M1-5	-	"	925	0.33	$3.2x10^5$	"
M1-6	-	29	121	0.16	$2x10^4$	"
M1-7	"	"	307	0.13	$4.2x10^4$	"
M1-8	-	-	427	0.13	$5.8x10^4$	"
M1-9	-	"	699	0.2	$1.5x10^5$	"
M1-10	-	-	961	0.17-2.9	$2.9x10^5$	"
M1-11	M1 shredded	35	1562	0.095	$1.48x10^8$	No reaction
M1-12	"	-	1666	0.095	$1.48x10^8$	"
M26-1	M26 paste	29	177	-	-	No reaction
M26-2	-	29	350	0.13	$9.4x10^7$	"
M26-3	-	29	800	0.35	$4.1x10^8$	"
M26-4	-	29	1666	0.31	$7.2x10^8$	"
M26-5	-	35	1600	0.09	$1.18x10^8$	Sparks[a]
M26-6	-	35	1612	0.02	$2.8x10^7$	"
RDX-1	RDX Slurry	29	769	0.02	$3.2x10^7$	No reaction
RDX-2		29	1724	0.11	$4.6x10^8$	"
RDX-3		35	1587	0.04	$5.29x10^7$	Sparks[a]
RDX-4		35	1562	0.07	$7.95x10^7$	"

[a]Probably metal sparks

Thermal Tests

Two types of thermal tests have been considered in the development of the hazards classification procedure. These are the local hot spot test and the regional thermal test. Before discussing these tests specifically, some background on thermal initiation problems will be presented. Three basic types of thermal initiation problems can be identified. These are illustrated in Figure 10 and discussed below.

External Oxidizer Required

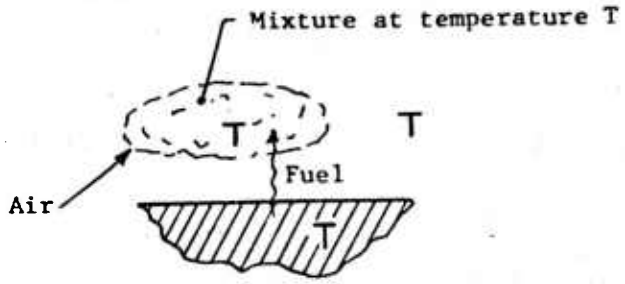
The first type of problem is where the fuel does not carry its own oxidizer. This includes open pools of flammable liquids such as hydrocarbon fuels, materials soaked in a volatile solvent, and situations involving a flammable gas-air mixture such as a leaking distillation tower. Ignition in this situation can occur in one of two ways: (1) an intense external ignition source (e.g., flame or spark) is inserted into a flammable fuel-air mixture or (2) the material is heated to sufficient temperature to produce a flammable fuel-air mixture and also ignite it. The first situation is characterized by the flash point test. The flash point temperature is the point at which the sample produces just enough fuel to form a flammable mixture. In the flash point test, the mixture is ignited by a pilot flame and the fuel is quickly consumed during the "flash" burn. The flash point test does not appear to be appropriate in hazards classification since a stable flame is present to cause the ignition. In this sense, the flash point test represents a secondary event.

The second situation is characterized by the autoignition test. The autoignition test exposes a sample to elevated temperatures in an air environment. The lowest temperature at which the sample is ignited (due only to the temperature) is the autoignition temperature in that it represents normal operations at elevated temperatures and cases where due to poor cleaning practices or an accidental leak or spill, the energetic material comes in contact with a hot surface (e.g., a hot motor casing or hot process vessel). Such situations are identical to that represented by the autoignition test.

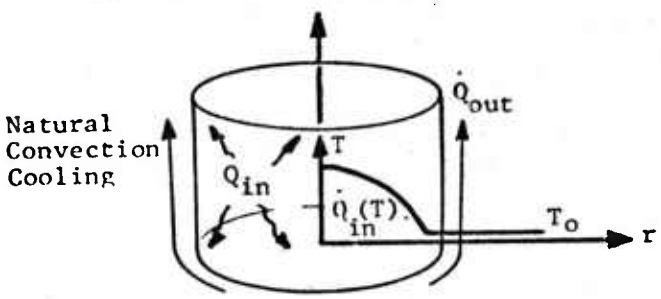
Runaway Reaction Within Static Mass of Material

The second category of problems involves a mass of energetic material which is believed to be stable and is for some reason brought to an elevated temperature, either locally or as a whole. One example of this problem is a mass of material in storage. The material could be a powder stored in a carton, a liquid in a drum or storage vessel, or a large propellant grain. Although the energetic material is stable at normal room temperatures, if the room's temperature is slowly increased, a point can be reached where chemical reaction within the material will proceed too quickly to be removed by conduction through the material

1. External oxidizer required
   Examples: hydrocarbon fuels, solvents, distillation processes with component failure



2. Runaway reaction within static mass of material
   Examples: explosive in storage, large propellant grain, liquid sitting in hold or storage tank, mix tanks, wash tanks, local hot spots, and radiant heating



3. Runaway reaction within an agitated, cooled fluid
   Examples: reactors, mix tanks, wash tanks

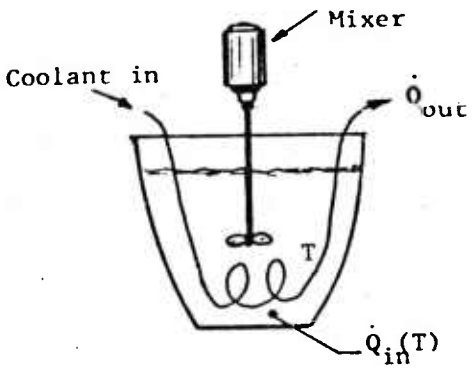


Fig 10 Categories of thermal initiation problems

and natural convection at the outer surface. When this occurs the critical temperature has been exceeded for the specific size of container involved and a runaway reaction will occur. This type of problem for simple configurations (e.g., cylinder, slab and sphere) is well characterized. If the material's thermophysical properties (density, heat capacity and thermal conductivity) and reaction rate properties (e.g., heat of decomposition, pre-exponential factor and activation energy) are known, the problem can be solved analytically introducing various approximations or more exactly using a computer model. For cases where internal heat transfer is present, the additional heat removal could be added to a simple computer model. In any case an estimate of critical temperature for a given container size can be made once the necessary material properties are known. Most of the required properties can be obtained from tests such as differential scanning calorimetry. These tests also provide the temperature at which the first significant exotherm will occur. Clearly, operation should be kept well below this value.

A second example of problems in this category is ignition by a local hot spot. This problem could also be solved analytically using a computer model, but in this case a realistic simulation could be done on a very small scale using a simple apparatus. A local hot spot test apparatus has been designed by IITRI personnel for this purpose.

Reactor Runaway Reaction

The third category of problems is perhaps the most difficult to be evaluated and has apparently not really been addressed in past hazards tests. A typical arrangement was illustrated in Figure 10. An exothermic reaction will proceed at a rate which is a function of temperature. As the temperature increases, the reaction rate increases. So that the process is well controlled, extreme care is put into assuring that sufficient heat transfer is available to maintain the system at its stable design temperature. Tremendous safety factors are inherent in these system designs. Nevertheless, accidental fires and explosions have occurred historically in this type of process operation. Incidents can be attributed to the following thermal causes:

- loss of cooling
- loss of mixing
- obstructed cooling or mixing allowing hot regions to develop
- reactants added too quickly to tank
- operation exceeds ignition temperature for a product of the reaction (including scale on pipe walls, etc.)
- reaction of contamination introduced into the system (e.g., piece of paper, oil, water, etc.)

When a loss of cooling, loss of mixing or a runaway reaction is sensed in a well designed system, deluge and/or dump is initiated in order to prevent a catastrophic incident. All of this indicates that most of the hazards associated with reactors and similar process operations should already be addressed by the system designers. In order to properly design the system, tests must be done to obtain calorimetric data (heat release rate as a function of temperature). Using this data the heat exchanger for the reaction is designed and the design should embody a tremendous safety factor. A hazards analysis should be done to identify failure modes leading to fire and/or explosion. The hazards analysis should result in a system designed to minimize the probability of an incident occurring. In other words, reactors are clearly hazardous unless properly designed. Hazards classification can make note of this for all reactors, but in the final analysis, safe design of reactors is really the designers responsibility and must be accomplished wherever a reactor is used.

In order to evaluate the thermal hazards of process operations in a relatively simple manner, two types of tests were selected. These are a localized thermal test and a regional thermal test. The experimental evaluations of these tests are described in the next two subsections.

Local Thermal Test

Inprocess explosive and propellant materials may be susceptible to initiation as a result of localized hot spots. The type of localized heating visualized here may be caused by a variety of sources, of which the following are examples:

- Friction sparks
- Welding sparks
- Hot metal chip caused by equipment fault
- Hot solid or liquid particles thrown from unplanned chemical reaction
- Lit cigarette

Since the causes of accidental initiation being considered are of a random nature, exposure of material samples to actual real-life stimuli of the types listed above would not be suitable for test purposes. First, these realistic stimuli cannot be reproduced or quantified easily. Second, too many tests would be required. A more practical approach is to devise a single type of stimulus which is representative of the above group, whose intensity can be varied over a wide range, is reproducible, and can be measured with fair accuracy.

The common characteristic of the modes of initiation being considered (viz. sparks, etc.) is that each consists of a particle at high temperature that delivers a small amount of heat to the host material over a small area. The heat comes from the cooling of the particle itself, and

the amount of heat can be estimated. To simulate this type of localized heating in a reproducible and measurable manner, one may consider a number of possibilities. These are described briefly in the following paragraphs.

Electrical Methods

Localized spot heating may be accomplished electrically either as resistance heating or by the discharge of a spark.

Resistance heating can be done by placing a resistance element in good contact with the surface of the test material and dissipating a limited amount of energy through the resistor in a short time. This is done usually by discharging an electric capacitor through the resistor. This method is applicable only to nonconducting materials. For use on electrically conductive solids or liquids, the resistor would have to be insulated electrically; but the thermal lag imposed by the insulation would negate the very conditions being simulated.

The resistor may be in one of the following forms:

- A single short resistance wire
- A thermister
- A strain gage

The above differ in their geometric configuration. The single wire is a line heat source and can be made in diameters as small as desired. It is relatively simple to construct, but its configuration does not resemble closely the heat sources being simulated.

The thermister may be in the form of a small bead only several thousandths of an inch in diameter. This would more closely resemble the typical shapes of the particles being simulated. A thermister has a high negative coefficients of electric resistivity and is normally used for measuring temperature. For our purposes, the semiconducting thermister bead would constitute the resistance element where the heat is generated.

A certain type of strain gage consists of a thin plastic substrate upon which is deposited a metal film which constitutes the resister. The resister may be an area less than 0.04 inches square. Both the thermister and strain gage resistor would merit further consideration for use on nonconducting materials.

A spark may be generated at the surface of a material by placing two conductors, in good contact with the surface, a small distance apart. If a sufficiently high voltage were imposed across the conductors, a spark discharge would bridge the gap between them. An electric discharge in this manner does not resemble the physical mechanism of the heat sources being simulated, and it is not known at this time whether or not there is an acceptable correlation between them.

Radiant Methods

Local hot spots may be imposed on a material as incident radiant energy, using either a laser beam or an incandescent lamp as the energy source. Either source would require optical focusing and a shutter system. The area of exposure would be controlled by the focusing lens, and the time of exposure by the shutter. Thus the intensity of heating and the total amount of energy delivered could be varied over a range.

Both of these methods, however, have certain characteristics that would make their choice for this application less than ideal. While the incident radiant flux would be highly reproducible, the fraction actually absorbed at the surface of a material would depend on the optical absorptance properties of the target material. Some of the materials, especially if in the liquid state, also may be partially transparent, in which case the incident energy would be absorbed in depth. This would deviate from the conditions being simulated and might lead to erroneous conclusions in the interpretation of test results.

Mechanical Methods

Local hot spots for test purposes may be generated by reproducing in as much as possible the real-life conditions that can cause this type of initiation — viz., the impingement of hot particles on the surface of the sensitive material. Two methods of doing this are described in the following paragraphs, one of which is recommended for adoption.

A direct way of imposing hot spots is to generate sparks and cause them to impinge on the test material. This may be done mechanically using an arrangement of components analogous to an ordinary cigar lighter. A small friction wheel could be rotated at a controlled uniform speed, and a sparking material would be pressed against the friction surface by a pre-set force. The extent of sparking produced with the arrangement would depend on the materials and on the applied force. The area and time of spark impingement on the sample could be controlled by interposing a screen between the spark generating apparatus and the sample. An aperture in the screen would govern the area of exposure, and the screen itself could serve as a shutter to control the time of exposure.

Although this arrangement is a realistic simulation of a material exposed to accident sparks, it has certain shortcomings concerning reproducibility and the uncertainty in quantifying the thermal exposure. It is not feasible to obtain single sparks; and even if it were feasible, there is no assurance that the thermal effect of one spark would be like that of the next. With a stream of sparks, there is a chance that two or more might strike an area sufficiently small for the thermal effects to be partially cumulative. In either case, it is impossible to describe the heating effect quantitatively since this would require a knowledge of particle size and initial temperature.

Another approach is to retain the realism of imposing a local hot spot with a hot particle, but to do so using one particle at a time

knowing both its size (mass) and initial temperature. This would involve preheating a particle to a uniform known initial temperature and then dropping it onto the surface of the sample material. The controlled variables would be the material of the particle, its size, and initial temperature--which together describes its initial heat content. Changes could be made by changing any or all of these variables. This method has the following advantages:

- Tests are highly reproducible, limited only by the ability to measure physical size and temperature.
- The heating effect is quantifiable in absolute terms.
- The method is applicable equally well to both solid and liquid materials.
- The method can be used on solids of different shapes, e.g., flakes, pellets, strands, etc.
- The method is independent of the electrical properties of the sample material.

This technique was selected for experimental evaluation in this project.

Test Apparatus and Procedure

The apparatus for imposing a hot spot on a sensitive material using a pre-heated particle consists essentially of a miniature high-temperature furnace as shown in Figure 11. With the tubular furnace in the upright position shown in the figure, a small steel ball of known mass is allowed to reach the steady-state furnace temperature. Then the entire furnace assembly is turned upside down so the heated ball can fall freely onto the test sample, and the results are observed.

The furnace consists of a ceramic tube around which is wound a resistance wire that can be heated electrically. A smaller ceramic tube with one end closed is inserted into the furnace tube and serves as a holder for the steel ball. This smaller tube is within an isothermal region of the furnace and extends to within about 1 inch of the furnace exit. It serves to guide the pellet during its fall and assures that the pellet will not strike the cooler end of the furnace tube during its exit. While at the bottom of the inner tube, the pellet is effectively completely surrounded by an isothermal enclosure and will attain the enclosure temperature in a short time.

The heating rate and temperature of the furnace are determined by controlling the voltage supplied to the heater using a variable voltage transformer. Furnace temperature is measured by a thermocouple inserted into the furnace tube from below until it contacts the bottom of the inner tube containing the pellet. The thermocouple is made of chromel and alumel wires protected by a double base ceramic insulator. The thermocouple is permanently mounted in the furnace. The steel ball is inserted into the furnace from the top. After the ball reaches the desired temperature, the sample is set in place to receive the hot ball.

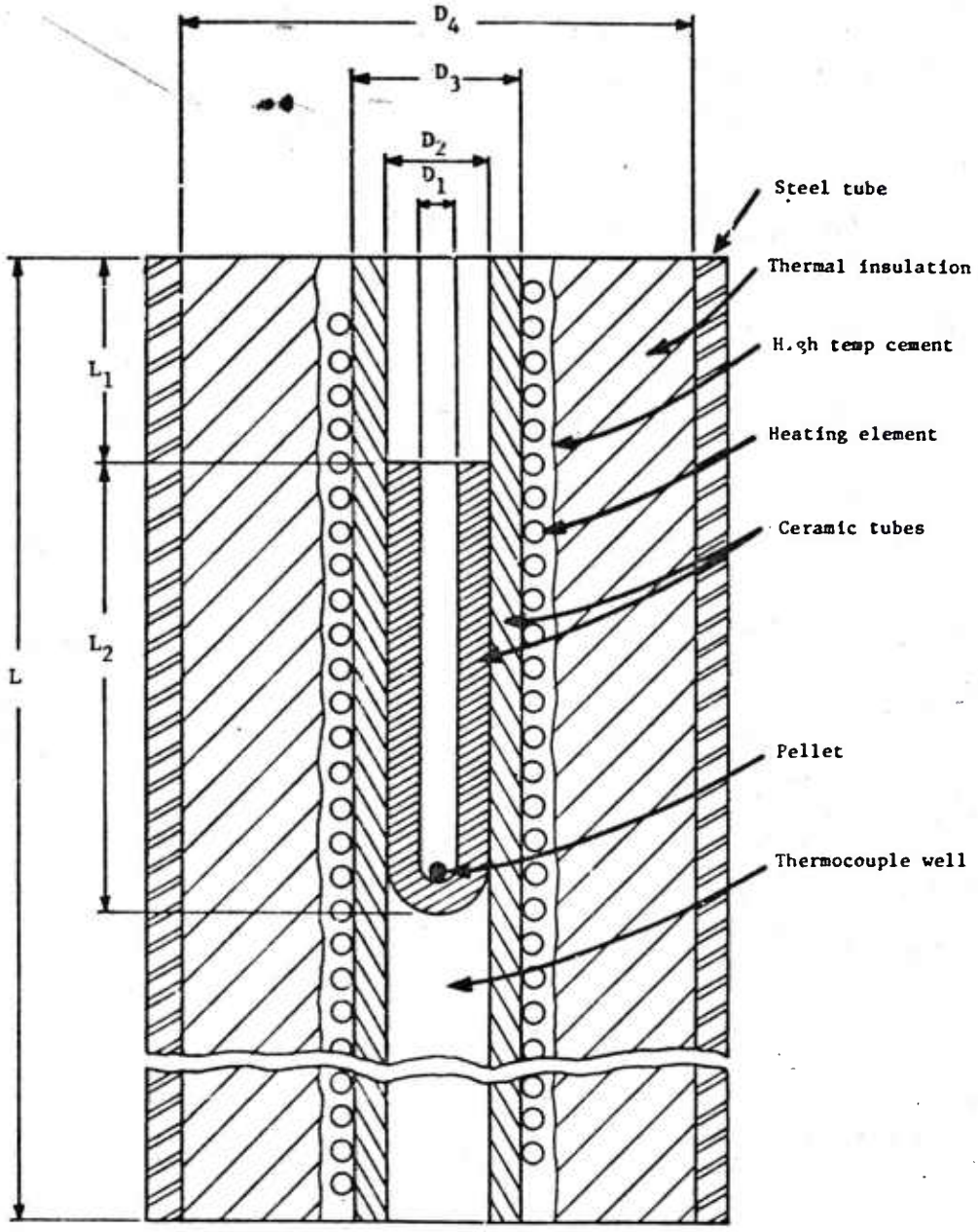


Fig 11 Furnace for heating small particles (not to scale)

The furnace tube is surrounded by high-temperature thermal insulation, and the assembly is enclosed by a steel tube to provide rigidity and protection for handling. The entire apparatus is so compact (Figure 12) that it can be supported readily on a small stand anchored rigidly on a bench (Figure 13). The mounting must be rigid so the pellet will strike the same spot each time it falls. Inversion of the furnace is done manually to a fixed stop that assured alignment of the exit tube with the sample.

Evaluation of Thermal Exposure

The test apparatus and thermal test procedure are such that the hot ball will experience little or no heat transfer during its fall to the surface of the sample material. If it is assumed that the heat transfer during the fall is zero, then the heating of the sample can be calculated readily. It would be the heat given off by the ball in cooling from the initial furnace temperature to the sample temperature (essentially ambient). The heat is given by:

$$q = mc(T_1 - T_2)$$

where m = mass of pellet

c = heat capacity of pellet material (specific heat)

T_1 = furnace temperature

T_2 = sample temperature

If the ball is of known material, its heat capacity will be known from the literature, and its mass and the two temperatures will be measured. Thus the heat given off will be known fairly accurately. Changes in the thermal exposure, if needed, will be known even more accurately since a change will be accomplished by using a ball of the same material but of different mass, or by changing its initial temperature.

Local Hot Spot Test Results

The local hot spot apparatus, test procedure, and sample loading are summarized in Figure 14. This test was found to be extremely simple and fast to accomplish; therefore, probit analysis was used to determine the experimental probability of ignition versus ball temperature relation. For hazards classification, the 50 percent probability of ignition point is needed. This is compared to the inprocess potential for the specific process operation being considered.

In this program only enough tests were completed to evaluate the test and to approximately determine the 50 percent probability point for each of the four sample materials.

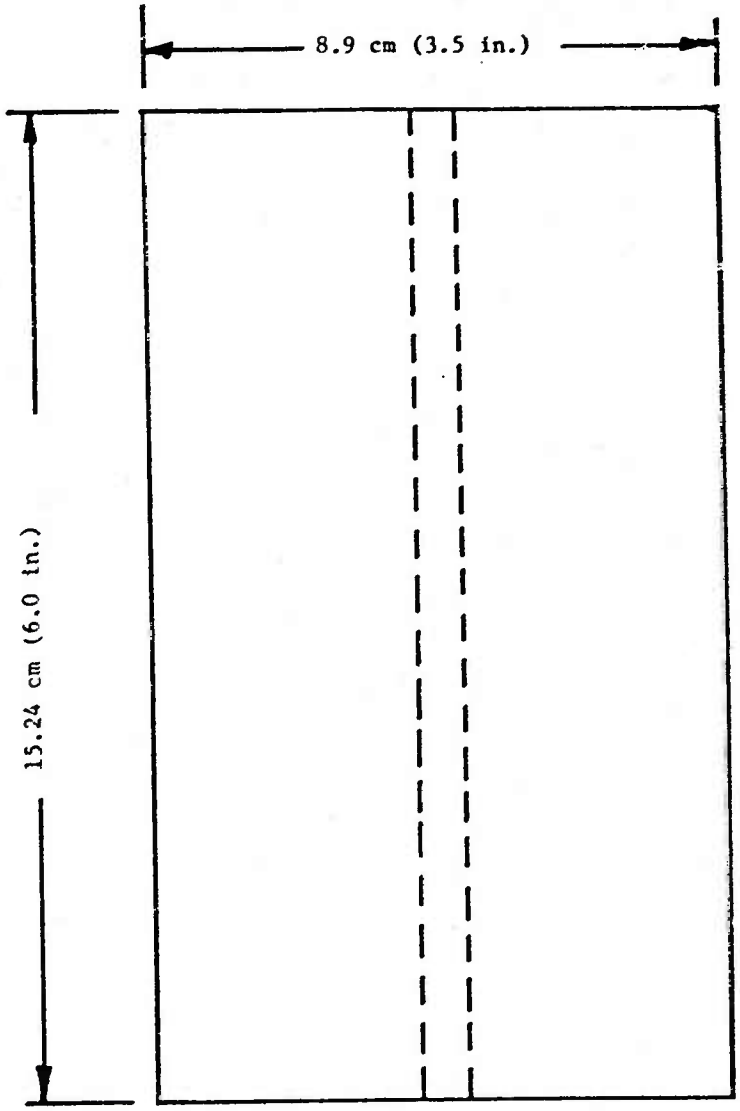


Fig 12 Actual size of furnace
(approximate outside dimensions

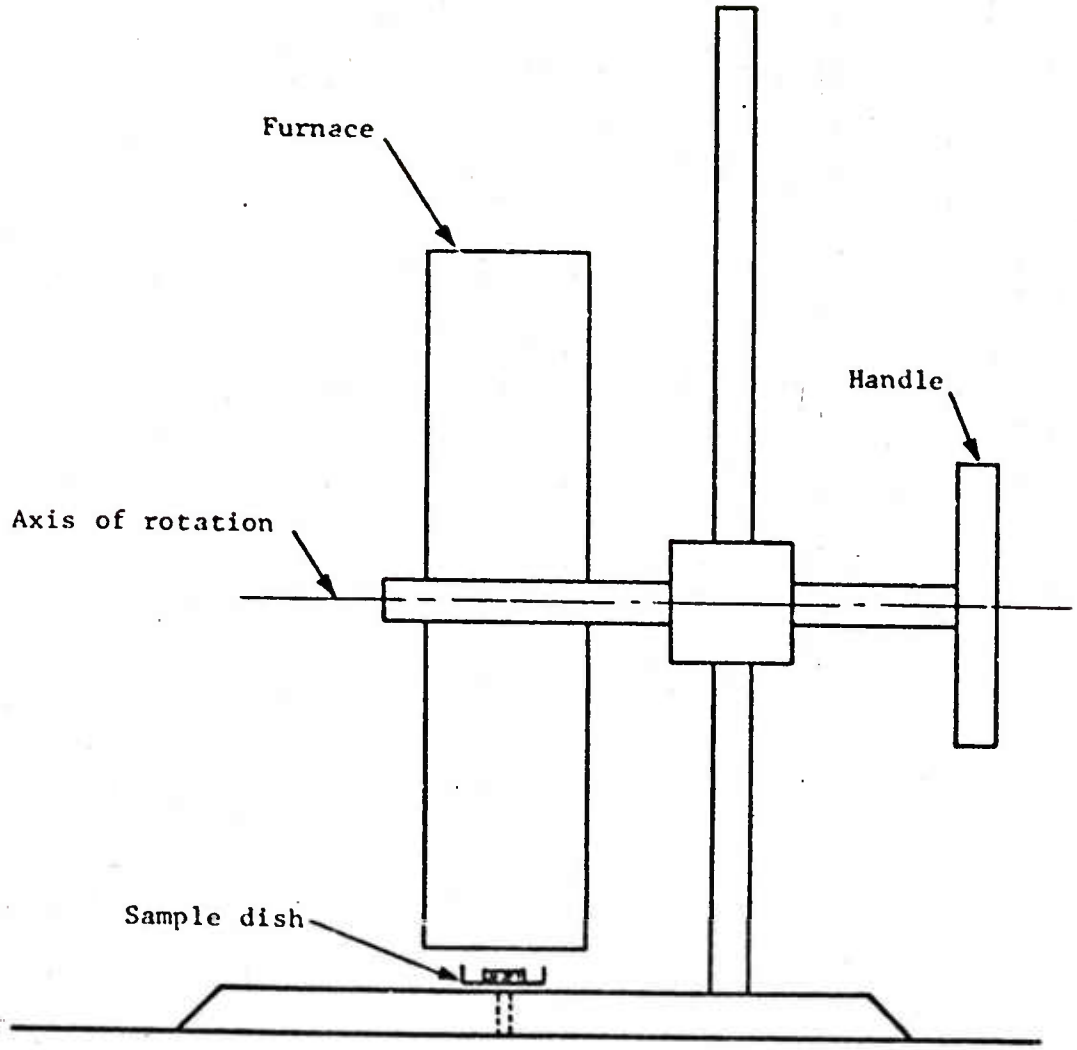
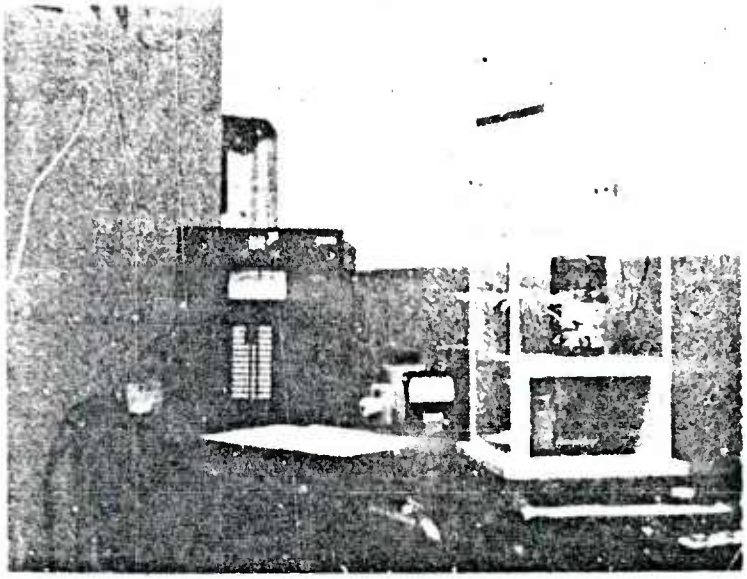
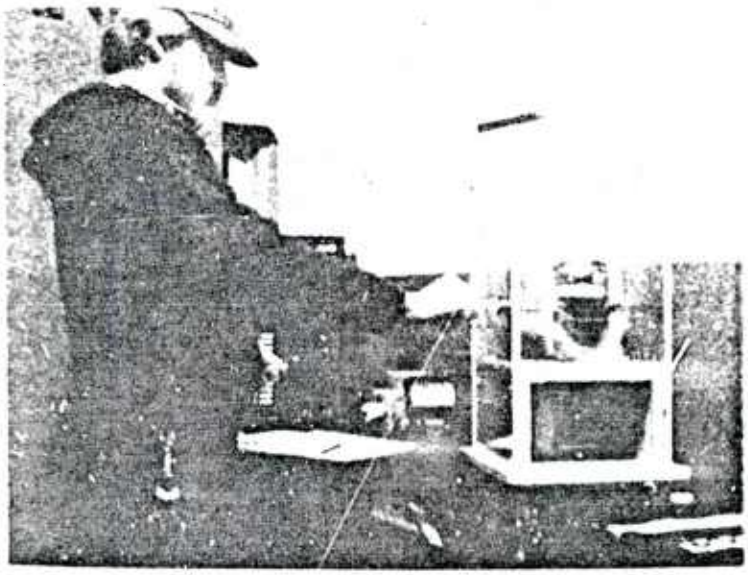


Fig 13 Setup for hot spot apparatus (not to scale)

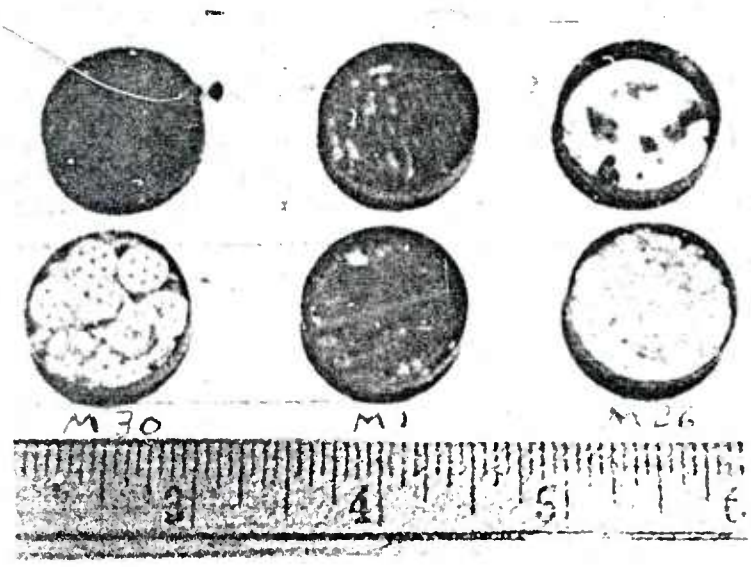


(a) Local hct spot test setup



(b) Test with M30 pellets (smoke only)

Fig 14 Local hot spot tests



(c) Test samples in sample holders

Fig 14 Concluded

Three ball sizes were tried in order to determine the best correlation parameter (i.e., ball temperature or ball energy). The three ball diameter were 0.079 cm (1/32 inch), 1 mm, and 0.119 cm (3/64 inch). The ball size appeared to have a negligible influence, whereas ball temperature dominated the results.

The local hot spot tests are summarized in Table 10 and Figure 15. As shown, the 50 percent probability ignition temperatures for the four materials were found to be:

Material	50 Percent Ignition Temperature, ^{o}C
M30 Pellets	~1035°C
RDX Slurry	>1066°C
M1 Strands	~786°C
M26 Paste	~500°C

Regional Thermal Test

Differential scanning calorimetry (DSC) was evaluated for hazards classification in the first program (Ref. 2). Most of this section has been extracted directly from the final report for that work.

Table 10

Local hot spot test results

Sample material	Ball temperature (°C)	Experimental probability of ignition	Comments
M 30 Pellets	510	0/2 = 0%	No reactions
	594	2/10 = 20%	Consumed, not burned
(T_{50}~1035°C)	788	3/15 = 20%	Burns
	1066	8/15 = 53%	Burns
RDX Slurry	510	0/3 = 0%	-
	788	0/8 = 0%	Wisps of smoke
(T_{50}>1066°C)	1066	0/15 = 0%	Wisps of smoke
M1 Strands	510	0/4 = 0%	
	705	5/5 = 50%	Burns
(T_{50}~786°C)	788	4/15 = 27%	Burns
	871	7/11 = 64%	Burns
	1066	10/10 = 100%	Burns
M26 Paste	233	0/2 = 0%	
	427	0/10 = 0%	
(T_{50}~500°C)	510	10/15 = 67%	Burns
	594	10/11 = 91%	Burns

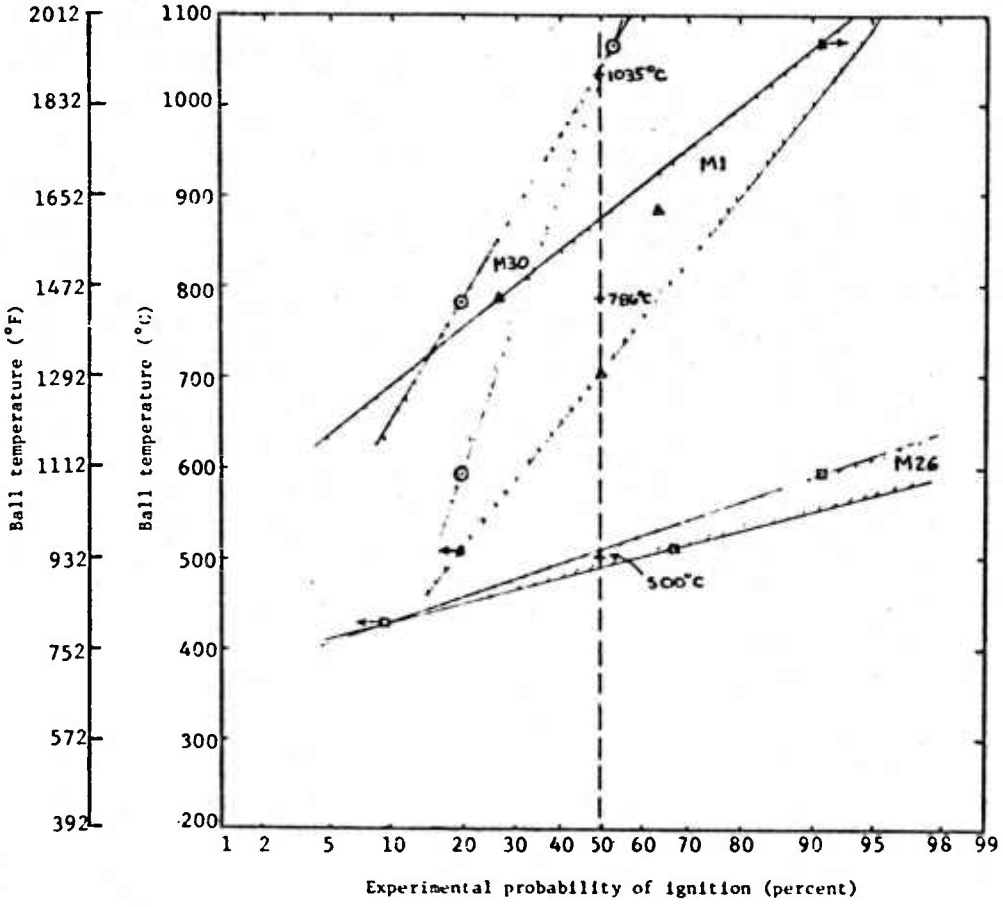


Fig 15 Local hot spot test results

The objective of this test is to determine the activation energy for inprocess materials and to determine the autoignition temperature corresponding to a given heating rate. Numerous process operations require heat addition to the working material, e.g., dryers, and melt-pour operations. This results in normal operating temperatures greater than ambient. Abnormal heat additions can occur due to failure of cooling equipment or steady frictional heating. The activation energy is a measure of a material's susceptibility to chemical decomposition at any temperature. A high activation energy indicates slow decomposition. The autoignition temperature places an upper bound for the safe operation of any process.

In the hazards classification system developed in the first project, of the four materials to be tested, only one (M30 pellets) represented an operation where the heating test would be necessary. The M30 air dried pellets are exposed to a drying operation which normally requires heat addition. Therefore only M30 pellets were comprehensively evaluated using DSC in that work. Data for RDX slurry was also obtained experimentally but the ignition temperature for M26 paste had to be estimated and for M1 strands had to be extracted from the literature.

Test Description

The DSC was chosen as the best method to achieve the desired objectives. During the test, the sample material and a reference material are heated simultaneously at the same rate. The sample and reference are contained in separate cups but placed in a common holder. Both cups are instrumented with thermocouples. The difference in electric power required to keep both sample and reference pan at the same temperature is recorded and the record is called a thermogram. An endothermic process (heat absorption) will require power to the sample pan and results in a downward deflection of the recorder pen. An exothermic process (heat release) will required less power to the sample and the recorder pen will deflect in the opposite direction. The temperature at which ignition occurs is clearly evident on the thermogram. The DSC analyses permit interpretation of phase changes, decomposition, melting points and thermal stability.

The test procedure is relatively easy. The tests were performed in atmospheric air. The instrument can handle other gaseous environments. A heating rate of 10°C per min was selected for these tests. The M30 sample was sliced to a size of 1 mm x 1 mm x 0.1 mm thick. A small lid was placed over the sample after it was placed in the sample cup. The reference used in these tests was merely an empty sample cup.

The thermogram for M30 triple base propellants (lot number RAD 77F0015012) is shown in Figure 16. Curve B is the M30 thermogram. Curve A is a blank run under the same conditions as the sample curve but with both pans empty. This was merely to confirm that a straight, horizontal base line was achieved. The M30 thermogram is seen to decrease monotonically as temperature increased between 40 and 170°C. This behavior

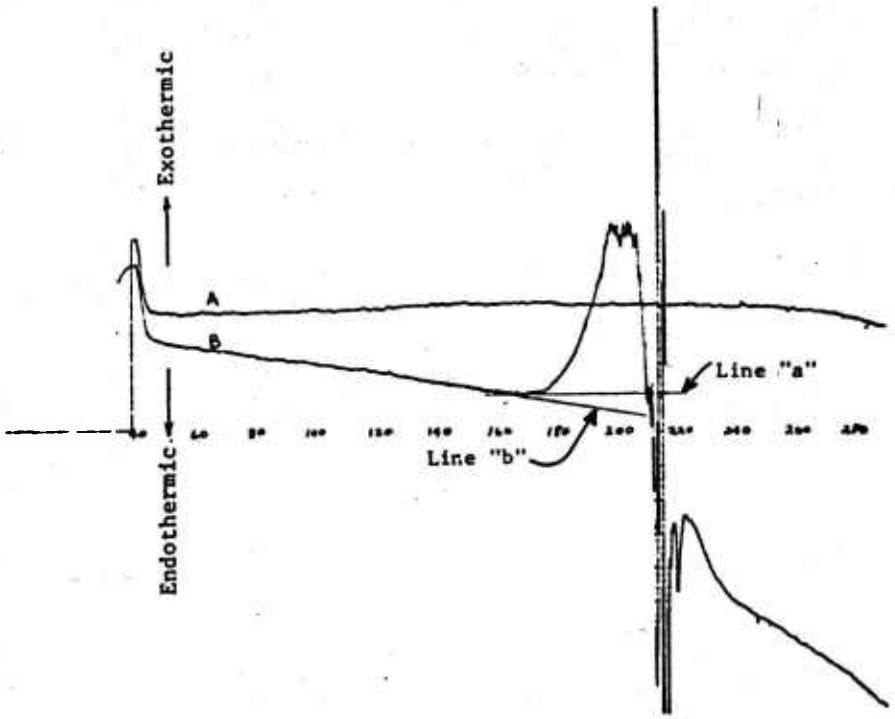


Fig 16 Example thermogram

is probably due to evaporation of the solvents that were present in the sample. At 170°C the M30 clearly begins to decompose thermally. This onset of an exothermic reaction leads to the autoignition of this material. Safe handling of the material should be limited to below 170°C.

RDX slurry was also evaluated using DSC. The autoignition temperature was determined to be 255°C.

Further analysis of the thermogram will produce the activation energy. The rate of energy evolution is proportional to the amount of pen deflection on the thermogram. In this case only the exothermic reaction is of interest. To measure the amount of deflection, it is necessary to select a base line. Two possibilities exist: choose a horizontal line, tangent to the peak of the endothermic reaction, or draw a line on an angle tangent to the endothermic curve. Both lines are drawn in Figure 16 identified respectively as line 'a' and line 'b', and the deflection was measured in arbitrary units from each base line up to the exothermic curve. The data are presented in Table 11.

Following the derivation and analysis methods of Ref. 14, the activation energy can be calculated from the following equation:

$$E^* = \frac{-19.16 \log_{10} (d_1/d_2)}{1/T_1 - 1/T_2} \quad \text{(J/mole)}$$

where d is the pen deflection at a temperature T. This also can be written:

$$E^* = -19.16m$$

where m is the slope of a straight line for log d versus 1/T. The data from Table 11 were plotted in Figure 17 to determine m. A low activation energy indicates rapid decomposition. Therefore, in analyzing the experimental data, it is safer to choose the method giving the lowest value of the activation energy. This indicates then, for M30 triple-base propellant pellets, the activation energy is 2.63×10^5 J/mole.

Relation Between Local and Regional Thermal Test Results

Figure 18 shows some Bureau of Mines data for black powder (Ref. 15). Three types of data are presented. The highest ignition temperatures correspond to ignition by small metal balls. The lowest ignition temperatures correspond to DSC results, in effect an infinite reservoir at the indicated temperatures. The transition between the local and regional ignition results was given by tests placing the sample on a large constant temperature surface for a specified duration. Based on this comparison of black powder thermal test results, it appears that the local hot spot test and the DSC realistically bracket the thermal ignition modes and should be adequate for hazard classification purposes.

Table 11

Thermogram data analyses for determining activation energy of M30 pellets

For straight horizontal baseline:

	d	T(°C)	T(°K)	$\log_{10} d$	$1/T \times 10^3$ (1/°K)
1	0.5	175	448	-0.3010	2.2321
2	2.0	180	453	0.3010	2.2075
3	5.5	185	458	0.7404	2.1834
4	10.0	190	463	1.0000	2.1598
5	17.5	195	468	1.2430	2.1367

$$m = \frac{\log_{10} (d_2/d_4)}{1/T_2 - 1/T_4} = \frac{-0.699}{0.0477} = -14.654$$

$$E^* = (-19.16)(-14.654) = 2.81 \times 10^5 \text{ J/mole}$$

For decreasing baseline (tangent to curve):

	d	T(°C)	T(°K)	$\log_{10} d$
1	1.2	175	448	0.0792
2	3.0	180	453	0.4771
3	7.0	185	458	0.8451
4	11.8	190	463	1.0719
5	19.2	195	468	1.2835

$$m = \frac{-0.9927}{0.0723} = -13.73$$

$$E^* = 2.63 \times 10^5 \text{ J/mole}$$

Horizontal baseline: $E^* = 2.81$ J/mole
Sloping tangent baseline: $E^* = 2.63 \times 10^5$ J/mole

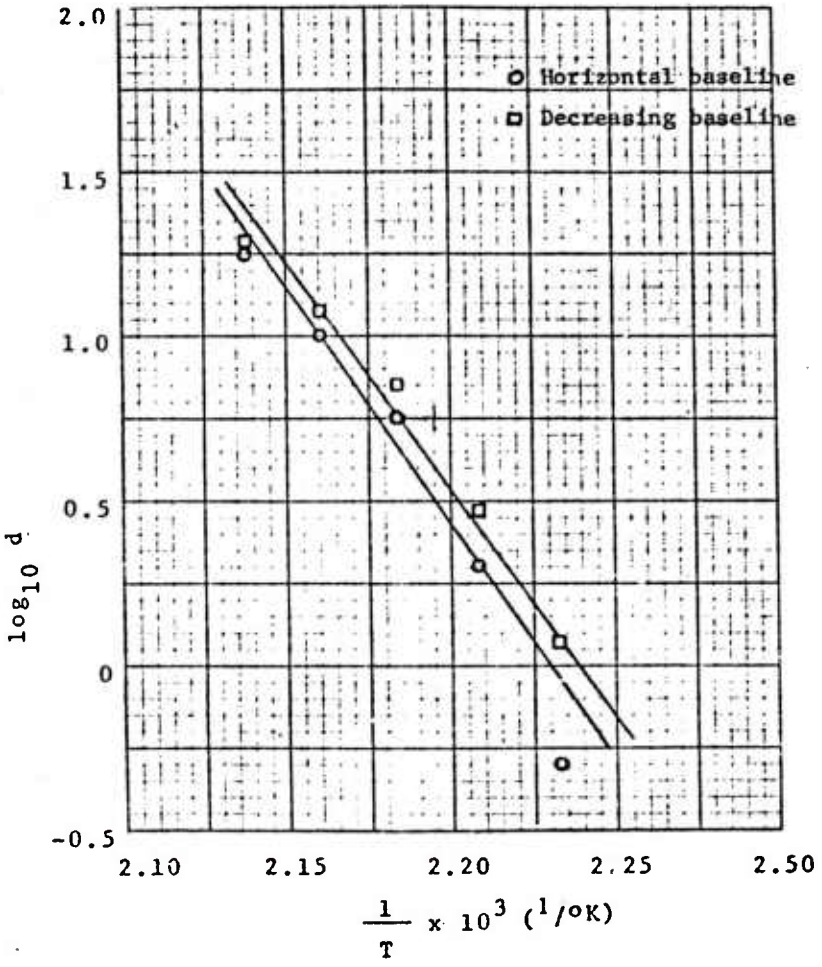


Fig 17 Determination of slope n for data in Table 11

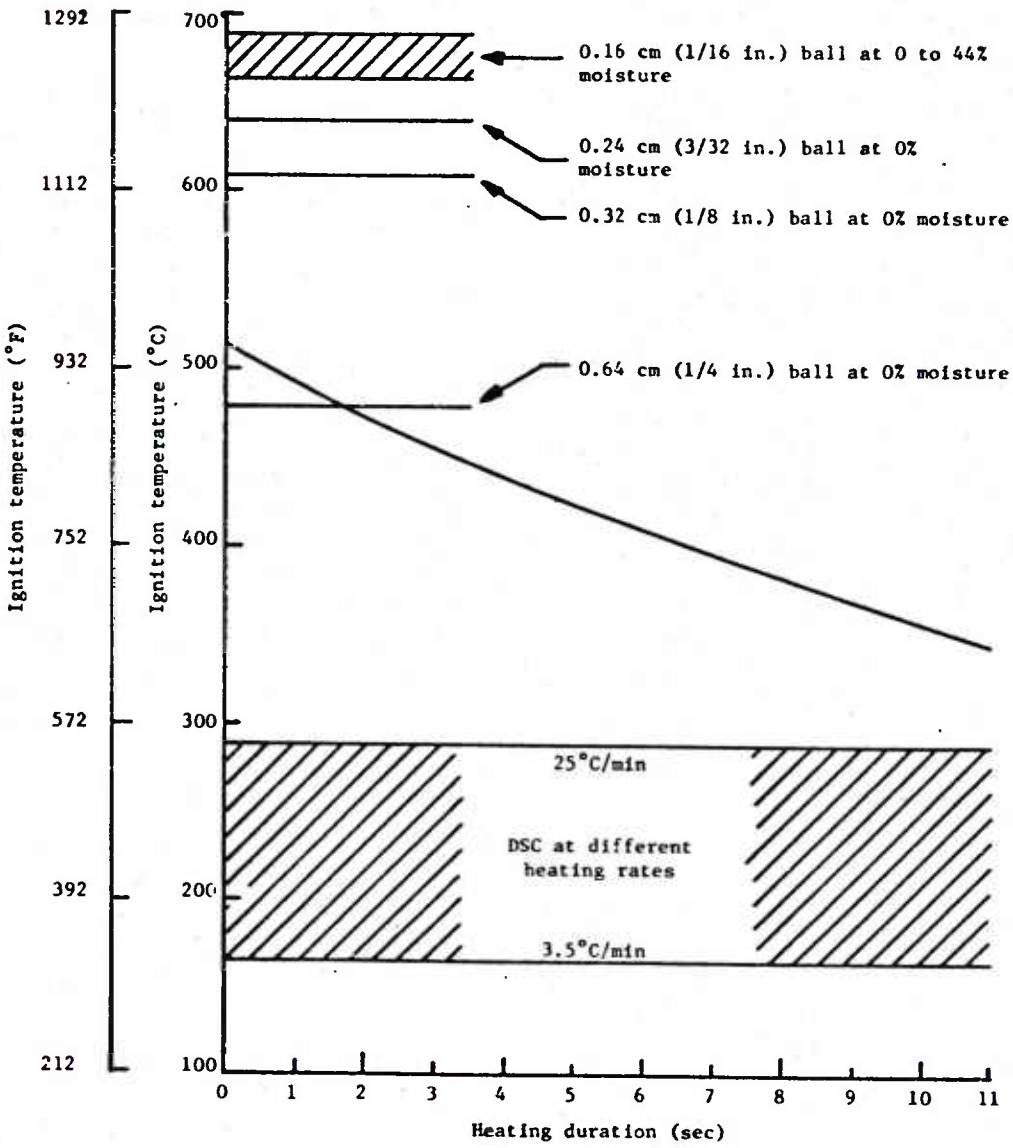


Fig 18 Thermal ignition of black powder (Bureau of Mines data)

Electrostatic Discharge Tests

In process plants, several types of electrostatic hazards exist. These include:

- charging of powder within sliding particle layers
- charging within dust or aerosol clouds inside vessels or unconfined
- charging within dielectric liquids
- ungrounded equipment items becoming charged
- ungrounded persons becoming charged
- dielectric surfaces becoming charged

The first three cases involve an exchange of electric charge between the process material and its surroundings. As the charge density within the material increases, the electric field intensifies until a discharge (charge relaxation releasing energy) occurs. If such a discharge has sufficient energy to ignite the process material, an energetic reaction (fire or explosion) will result.

The last three electrostatic hazards listed above involve something other than the process material becoming charged. When such an item becomes charged, its voltage increases. If the item's voltage becomes high enough to cause a discharge to occur, generally to a nearby grounded item, and if the process material is exposed to the discharge, ignition can occur. A person can store on the order of 15 millijoules of electrostatic energy and ungrounded metal items can easily store several joules. Again, if the discharge energy is sufficient to cause ignition, a fire or explosion can result.

As can be seen from the above discussion, the electrostatic hazard reduces two characteristics: the susceptibility of the process material to become electrically charged and the ignition sensitivity of the material to an electrical discharge. The problem is actually somewhat more complex in that different material will discharge ("breakdown") at different electric field strengths, but for purposes of hazards classification the simple view of the hazard in terms of charging susceptibility and ignition discharge energy will be adequate.

Charging susceptibility is characterized by the electrical relaxation time of the process material. A discussion of the charging mechanisms and formulation of an equation for charge density being proportional to relaxation time are presented in Reference 21. The electrical relaxation time of a material is equal to the ratio if the material's permittivity to its conductivity, both quantities of which are measureable. To characterize the ignition sensitivity of a material to electrostatic discharge, the sample must be exposed to discharges of various energy levels in a manner analogous to most of the other types of sensitivity tests.

A comprehensive experimental evaluation of the available measuring techniques for the electrical properties (permittivity and conductivity) was completed under the prior hazards classification contract. Small sample holders (typical of the sizes generally used for these tests) were used. The samples were generally pressed and did not represent the real process form. It was decided to improve these tests in the present program by allowing the sample to be placed in the sample holder at the same bulk density as it exists in the actual process. For inhomogeneous samples, such as pellets and strands, this required that the sample holder be made significantly larger.

In the paragraphs below, the techniques which have been used to measure the different electrical properties of the test samples will be described. These include discussions of permittivity, conductivity, relaxation time, and ESD ignition energy.

Permittivity

The permittivity, ε, is usually expressed as the relative permittivity, ε_r, with respect to the permittivity of free space, ε_o.

The relative permittivity is,

$$\varepsilon_r = \frac{\varepsilon}{\varepsilon_o}$$

and is referred to as the dielectric constant, K. The permittivity of free space has a value

$$\varepsilon_o = \frac{1}{36 \times 10^9} = 8.85 \times 10^{-12} \quad (\text{coul}^2/\text{n-m}^2)$$

The dielectric constant of a material can be determined by measuring the influence of the test material on the capacitance of a parallel plate condenser. A condenser is formed wherever an insulator (i.e., dielectric) separates two conductors between which a difference of potential can exist.

In the case where the condenser electrodes are plates having a constant spacing, the capacitance, C, is given by the expression

$$C = 0.08842\ k \frac{A}{d} \qquad (\text{pf})$$

where

A = area of active dielectric in square centimeters

d = spacing between plates in centimeters

K = dielectric constant

The dielectric constant is a material property and is substantially independent of frequency unless polar effects are involved.

Molcules of a dielectric may be either polar or nonpolar. For polar molecules, the dielectric constant under alternating-current conditions is increased as a result of the rotation of the polar molecules under the influence of the applied voltage (Ref. 20). The extent to which this polar action is effective depends upon the frequency and the temperature. If the temperature is lowered sufficiently, polar rotations are prevented, causing the dielectric constant of the material to drop. Similarly, if the frequency is made sufficiently high, the polar molecules are not able to follow the alterations of the applied field and the dielectric constant drops. The losses exhibited by dielectrics appear to be associated with the presence of polar molecules and free ions.

Polarization and conduction are the cumulative results of molecular charge carrier movement in the dielectric material. Polarization involves the action of induced dipoles. Conduction refers to the number of free charge carriers (electrons) persent. When an alternating-current is applied, the dipoles oscillate because of the cyclic nature of the electric field. The dipole oscillation stores and dissipates the energy. Accordingly, the dielectric properties of the material can be expressed in terms of the dielectric constant and the loss factor. The dielectric constant is related to the amount of electric field energy that the dipoles in the material temporarily store and release during each half cycle of the electrical field change. The loss factor expresses the dissipation of energy caused by both conduction and dipole oscillation losses. The dielectric constant and loss factor are expressed in combined form as a complex permittivity

$$\varepsilon_r = \frac{\varepsilon'}{\varepsilon_o} - j\frac{\varepsilon''}{\varepsilon_o}$$

where

ε_r = complex dielectric constant

ε' = dielectric constant

ε'' = loss factor

j is the phasor operator $(\sqrt{-1})$

Permittivity Measurements

The dielectric constants of the inprocess propellant materials were obtained by measuring the effect of the material on the capacitance of a parallel plate condenser. Figure 19 is a block diagram of the test setup used for these determinations.

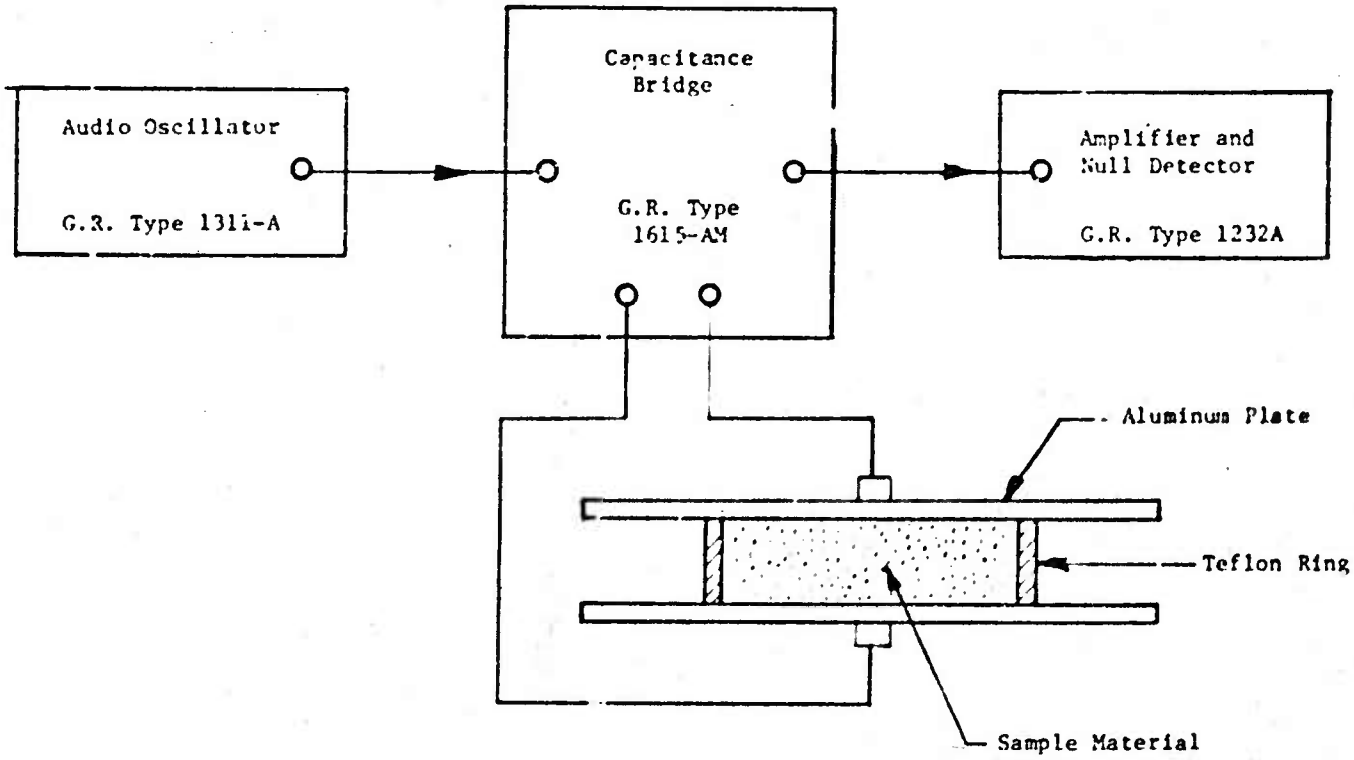


Fig 19 Block diagram of test configuration for the permittivity test

The parallel plate capacitor consists of two aluminum disks 45.72 cm (18.0 inch) in diameter and 2.54 cm (1.0 inch) thick. Copper leads 0.0794 cm (0.0312 inch) by 0.635 cm (0.25 inch), approximately 45.72 cm (18.0 inch) long were used to connect the parallel plates to the measuring equipment. The measuring equipment consisted of a General Radio Company Type 1620-A capacitance measuring assembly; a Type 1311-A audio oscillator, a Type 1615-AM capacitance bridge, and a Type 1232-A tuned amplifier and null detector.

The teflon ring was used to contain the sample in a cylindrical shape and also to maintain the parallel plate spacing. Three sizes of teflon rings were used for various tests. The purpose for using three ring sizes was to obtain independent measurements for assessing the precision of the measurements. Permittivity is a material property and therefore should be independent of dimension. The three teflon rings had equivalent diameters, 23.50 cm (9.25 inch) ID and 24.13 cm (9.50 inch) OD. The three height dimensions were 0.635 cm (0.25 inch), 1.27 cm (0.50 inch) and 1.91 cm (0.75 inch).

The parallel plate capacitor described here is relatively large. The purpose of providing a large sample holder was to accommodate the propellant materials in their inprocess form. We believe it is important to test the material with a minimum amount of preconditioning of the test samples.

The technique for determining the diclectric constant of the materials was patterned after those described by E. E. Walbrecht (Ref. 8). With no sample in the fixutre, the capacitance was measured by separating the plates a known distance. Three small teflon disks were used for this purpose. The disks were 0.635 cm (0.25 inch) dia. by 0.635 cm (0.25 inch) long and were used to simulate the separation distance provided by the smallest, previously described, teflon ring. The measured capacitance was compared with the calculated value. The difference is attributed to stray capacitance and fringing. The influence of this capacitance was accounted for by calculating an effective plate area. The effective plate area has a value that would provide a capacity equal to the measured capacitance. Finally, these procedures were repeated to determine the effective plate area for the 1.27 cm (0.50 inch) and 1.91 cm (0.75 inch) separation distances.

The teflon ring holder was placed at the center of the aluminum disk and filled with the test material. The second aluminum disk was placed on top of this assembly as shown in Figure 19. The measured capacitance, C, is the sum of capacitance contributions from the air area, teflon holder area and sample area,

$$C = \frac{\varepsilon_o}{d} \left(K_o A_a + K_1 A_c + K A_s \right)$$

and

$$K = \frac{Cd}{\varepsilon_o A_s} - \frac{(K_o A_a + K_1 A_c)}{A_s}$$

where

K = dielectric constant of sample

K_1 = dielectric constant of ring holder ($K_1 = 2.1$)

K_o = dielectric constant of air ($K_o = 1.0$)

d = plate separation (cm)

A_a = effective cross-sectional area of air area (cm^2)

A_c = ring holder cross section area (cm^2)

A_s = sample cross section area (cm^2)

Conductivity

The conductivity of the material, σ, is the reciprocal of resistivity, ρ. The procedure for determining conductivity of the material is to measure the sample's resistance, R. Using the measured value of R and the physical dimensions of the sample, the conductivity is determined:

$$\sigma = \frac{d}{A_s R} \qquad \text{(mho/cm)}$$

where A_s and d are the cross-sectional area and length of the sample, respectively.

The instrument used for the resistance measurements was a Hewlett Packard Model 4329A High Resistance Meter. This unit has a range 500K ohm to 2×10^{16} ohm and can be used with 7 test voltages in the range 10 volts to 1000 volts. The capability for varying the test voltage is useful for identifying voltage coefficient of the materials. Unfortunately, the relatively high conductivity of the M26 and M30 materials did not permit these measurements with the existing equipment.

An alternate method for determining conductivity was a direct measure of conductance, G. This method provides a means for obtaining conductivity as a function of frequency. The equivalent circuit and phasor diagram for this determination is shown is Figure 20.

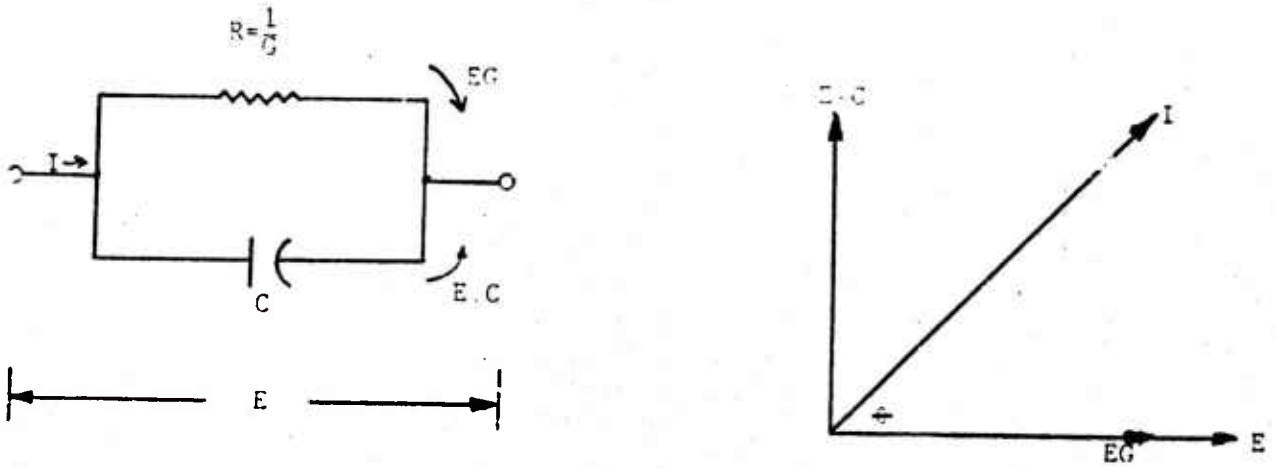


Fig 20 Equivalent circuit and phasor diagram for conductance test

Relaxation Time Constant

The ability of a material to store electrostatic charges is related to the relaxation time, τ. If the relaxation time is short, charges will be dissipated as fast as they are acquired. If it is long, more charges will be acquired than lost and the electtic charges will build up on the surface of the material. The relaxation time constant for a material can be calculated

$$\tau = \varepsilon/\sigma \qquad \text{(seconds)}$$

where ε is the permittivity and σ is the conductivity. For a particular material sample

$$\tau = RC = \rho \frac{d}{A_s} \times \varepsilon_o K \frac{A_s}{d} = \rho \varepsilon_o K = \rho \varepsilon \qquad \text{(seconds)}$$

Energetic Materials

The energetic materials tested during the program were M1 strands, M30 pellets, M26 paste and RDX slurry. Preconditioning of the test samples was nominal and did not significantly influence the measurement. The M1 strands were cut to lengths of approximately 2.5 cm (1.0 inch) in order to obtain the desired packing density in the test fixture. The M30 pellets were placed in the test fixture in an orderly fashion, rather than random packing. The M26 material on hand showed considerable agglomeration; however, granular samples were selected for the tests.

Ideally, the sample density for the electrical tests would have been the same as those used for the previously conducted sensitivity tests (i.e., 0.838 gr/cm^3 for M30, 0.829 gr/cm^3 for M26 and 0.45 gr/cm^3 for the M1 samples). These densities were not maintained for the electrical properties test because the parallel plate test fixture could not support the required pressing force. The procedure used here was to brim fill the sample holder, thus assuring electrical contact with the plates while maintaining a constant separation distance. The test samples were weighed and the density calculated after the electrical measurements were completed.

Experimental Data

The experimental data is presented here in tabular form. The permittivity, resistivity and relaxation time are shown as a function of the test frequency. Table 12 contains the results of the M1 material test. As shown in Table 12, the electrical properties of inprocess M1 material are frequency dependent. The permittivity, resistivity and relaxation time show a pronounced increase with decreasing frequency. The permittivity is in the range $15 - 40 \times 10^{-12}$ coul^2/N·M^2 and the resistivity is in the range $0.5\text{-}15 \times 10^9$ ohm-meter. The relaxation time is in the 10^{-2} to 10^0 seconds range.

Table 12

Electrical properties of inprocess M1 materials

Sample M1-1 Density = 0.353 gr/cm^3 Plate separation, d = 0.541

	Frequency (kHz)							
	0.05	0.1	0.2	0.5	1.0	2.0	5.0	10.0
Permittivity, $\varepsilon = K\varepsilon_o$ (coul^2/N·M^2 x 10^{-12})	20.5	20.1	18.6	17.2	16.4	15.0	15.1	14.7
Resistivity, ρ (ohm-meter x 10^9)	16.0	7.20	4.25	4.01	3.37	3.37	3.21	2.72
Relaxation time, τ (seconds x 10^{-3})	328	145	79	69	55	53	49	40

Sample M1-2 Density = 0.353 gr/cm^3 Plate separation, d = 1.924 cm

	Frequency (kHz)							
	0.05	0.1	0.2	0.5	1.0	2.0	5.0	10.0
Permittivity, $\varepsilon = K\varepsilon_o$ (coul^2/N·M^2 x 10^{-12})	38.4	33.7	29.6	25.4	23.2	21.8	20.1	19.2
Resistivity, ρ (ohm-meter x 10^9)	6.57	1.29	0.70	0.64	0.64	0.66	0.53	0.53
Relaxation time, τ (seconds x 10^{-3}	252	44	21	16	15	14	11	10

Sample M1-3 Density = 0.353 gr/cm^3 Plate separation, d = 1.344 cm

	Frequency (kHz)							
	0.05	0.1	0.2	0.5	1.0	2.0	5.0	10.0
Permittivity, $\varepsilon = K\varepsilon_o$ (coul^2/N·M^2 x 10^{-12})	42.0	33.6	27.2	25.7	23.2	22.0	20.1	19.0
Resistivity, ρ (ohm-meter x 10^9	5.91							
Relaxation time, τ (seconds x 10^{-3})	248							

The M26 material also shows an increase in electrical properties with decreasing frequency, Table 13. The permittivity is in the range 60 - 300 x 10^{-12} $coul^2/N \cdot M^2$ and the resistivity is about 2 - 7 x 10^5 ohm-meter. The relaxation time is about 1 - 10 x 10^{-5} seconds.

The M30 sample data is shown in Table 14. The elctrical properties are similar to those described for M26. The relatively large pellet size for the M30 material was not compatible with the dimensions of the sample holder. Accordingly, only one sample size of M30 material was evaluated.

Table 15 summarizes the results for the RDX slurry. The permittivity was measured between 20 - 40 $coul^2/N \cdot M^2$ and resistivity was measured between 8 and 85 x 10^6 ohm-meter. Therefore, within the range of measured values, the relaxation time was found to be about 2 to 33 x 10^{-4} seconds.

Observations and Comments

The materials tested appear to behave as polar dielectrics. This tendency is manifested by the substantial increase in permittivity for decreasing test frequency.

The relatively low values of resistivity for inprocess test samples (i.e., compared to pressed samples), indicates that the solvent content may be the overriding factor in determining the electrical properties of the materials in their inprocess form. It appears that the physical characteristics of the material should be well defined for a particular process stage. The electrical properties could then be assessed for these conditions. Testing preconditioned samples is perhaps a more satisfying task; however, this technique provides little information on the inprocess characteristics.

The requirement for assessing the electrical properties at d.c. (i.e., test frequency = 0) is clear. The increase in permittivity, resistivity and relaxation time with decreasing frequency suggests that larger values of relaxation time will occur at or near d.c. Also, the mechanism for the material to acquire charges in process plants is due to relative motion between the material particles and the container wall. One could reasonably assume that these charges would be acquired at a low rate -- analogous to a low frequency.

Figure 21 shows the measured electrical relaxation time plotted against frequency on log-log paper. In all cases except for M1 strands, the data seems to be leveling off at low frequencies. If we arbitrarily select a low frequency of 1 per second in order to characterize the materials susceptibility to charging, the data in all cases can be approximately extrapolated back to that value to define the material's "low frequency" relaxation time. For M30 pellets, the relaxation time is about 1.3 milliseconds. For M26, the time is about 0.2 milliseconds. For RDX slurry, the time is about 25 milliseconds. For M1 strands,

Table 13

Electrical properties of inprocess M26 material

Sample M26-1 Density = 0.571 gr/cm^3 Plate separation, d = 1.344 cm

	Frequency (kHz)						
	0.05	0.1	0.2	0.5	1.0	2.0	5.0	10.0
Permittivity, $\varepsilon = K\varepsilon_o$ (coul^2/N·M^2 x 10^{-12})		301	262	206	173	157	114	95
Resistivity, ρ (ohm-meter x 10^6)		0.27	0.26	0.25	0.25	0.25	0.20	0.16
Relaxation time, τ (seconds x 10^{-6})		81	68	52	43	39	22	15

Sample M26-2 Density = 0.527 gr/cm^3 Plate separation, d = 1.984 cm

	Frequency (kHz)						
	0.05	0.1	0.2	0.5	1.0	2.0	5.0	10.0
Permittivity, $\varepsilon = K\varepsilon_o$ (coul^2/N·M^2 x 10^{-12})		163	140	120	106	90	72	61
Resistivity, ρ (ohm-meter x 10^6)		0.68	0.68	0.66	0.64	0.61	0.46	0.35
Relaxation time, τ (seconds x 10^{-6})		111	95	79	67	55	33	22

Table 14

Electrical properties of inprocess M30 material

Sample M30-1 Density = 0.942 gr/cm^3 Plate separation, d = 1.502 cm

	Frequency (kHz)							
	0.05	0.1	0.2	0.5	1.0	2.0	5.0	10.0
Permittivity, $\varepsilon = K\varepsilon_o$ (coul^2/N·M^2 x 10^{-12})		236	211	180	160	137	112	94
Resistivity, ρ (ohm-meter x 10^6)		4.74	4.57	3.15	2.05	1.36	0.66	0.38
Relaxation time, τ (seconds x 10^{-6})		1120	962	566	328	127	74	35

Table 15

Electrical properties of inprocess RDX material

Sample

	Frequency (kHz)							
	0.05	0.1	0.2	0.5	1.0	2.0	5.0	10.0
Permittivity, $\varepsilon = K\varepsilon_o$ (coul^2/N·M^2 x 10^{-12})	38.3	37.3	30.9	28.0	27.1	26.6	25.4	24.7
Resistivity, ρ (ohm-meter x 10^6)	84.3	62.5	50.0	37.0	30.3	20.8	13.9	8.4
Relaxation time, τ (seconds x 10^{-6})	3229	2331	1545	1036	821	553	353	207

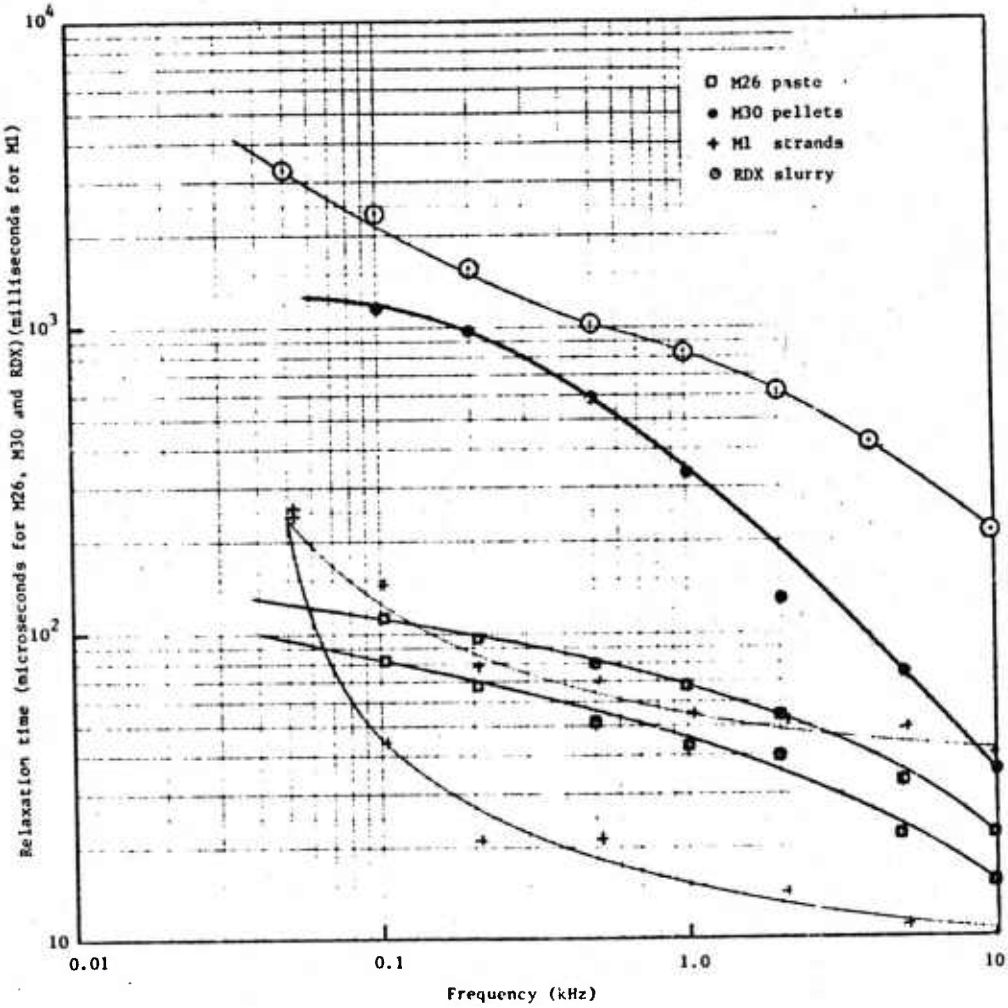


Fig 21 Relaxation time versus frequency for the inprocess materials tested

the relaxation time is about three hours. By comparing these results with a time (say 0.1 seconds) which should characterize the charging process of for example particle-wall contacts, M1 is found to be highly susceptible to charging, M30 is not susceptible to charging, M26 is not susceptible to charging, and RDX is marginal.

The use of a relatively large sample holder to test inprocess materials provides a feasible approach. The relatively large size of the M30 pellets required orderly packing — as opposed to random packing. However, the M30 materials can be tested in their inprocess form using this procedure.

Finally, it should be noted that the data reported here was obtained under field conditions. It is anticipated that the precision of measurement could be improved with improved test procedures. Controlling and/or determining the solvent content of the test material would provide useful information. Also, the control of external factors, such as temperature and relative humidity would improved the quality of the measurements.

ESD Ignition Tests

The circuit used to conduct the ESD ignition tests is shown in Figure 22. The capacitor is charged to the desired voltage level, V. The energy stored in the capacitor is given by

$$e = \frac{1}{2} CV^2$$

where C is the capacitance. Not all of this energy will be dissipated in the discharge. For this reason, the resister R_1 is added to the circuit. Measuring the voltage across this resistor as a function of time $V_1(t)$ gives the current-time relation through the sample

$$i_s(t) = \frac{V_1(t)}{R_1}$$

The voltage across the sample is obtained from the difference between the two measured voltages:

$$V_s(t) = V(t) - V_1(t)$$

The energy dissipated in the discharge in the sample is then given as the integral over time of the power $(V_s(t) \cdot i_s(t))$:

$$e = \int_0^\tau V_s(t) \cdot i_s(t)\, dt$$

where τ is the discharge duration.

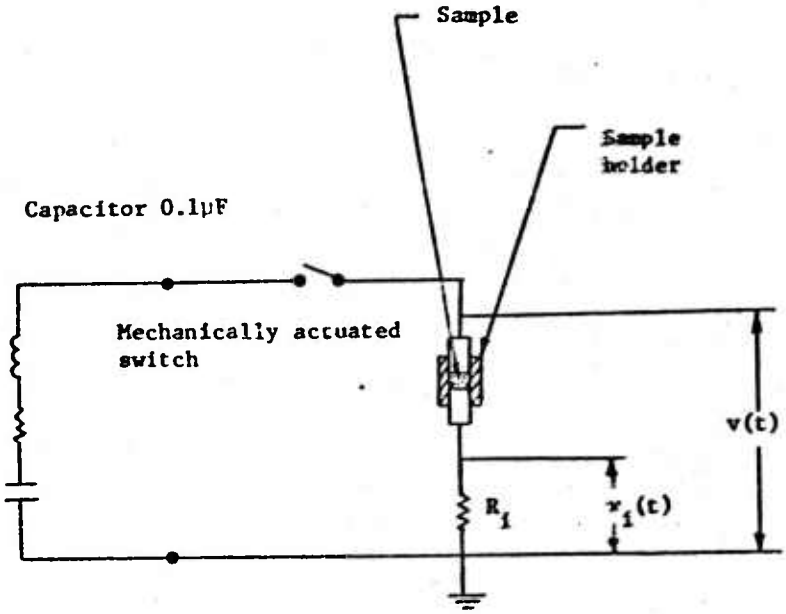


Fig 22 Schematic circuit diagram for electrostatic discharge test

The sample holders used for powdered and pellet or strand materials are shown in Figure 23.

For powder samples, the spark gap was always 0.32 cm (1/8 inch). For pellets and strands, the needle electrodes were always on opposite sides of the sample along the circumference. More orientations of the electrodes should have been tried in the testing, but only a limited number of tests could be accomplished. It is expected that the discharge energy will have a different effect for different gaps between the electrodes. Therefore, the energy probably should be expressed as energy per unit spark length. Spark length will be the gap length for powder or liquid samples and will be the distance between the electrode needle tips along the circumference for pellet or strand type samples. For the final hazards classification procedure, it is suggested that three gaps (0.318 cm, 0.155 cm, and 0.079 cm) be used as a standard. In the future, more work should be done to better characterize the relationship between spark gap and ignition potential in order to possibly reduce the number of tests required.

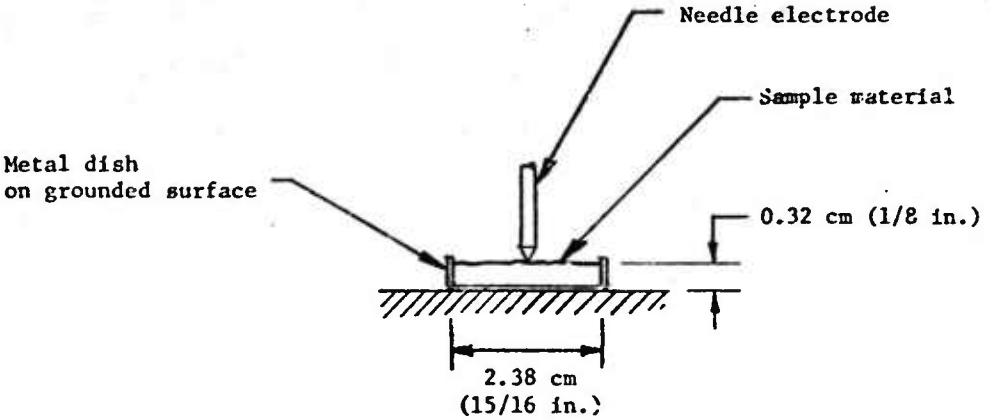
The ESD tests which were conducted are summarized in Table 16. No initiations were obtained in any of the tests which were conducted. For M30 pellets in 4 out of 15 trials (27 percent) burn marks were observed when 5 joules stored at 10 kilovolts in a 0.1 microfarad capacitor were discharged into the sample. From the oscilloscope records, it was found that only between 7 and 32 percent (typically 18 percent) of the capacitor energy is actually dissipated in the sample. Therefore, the discharge energy experienced by the sample was only about 1 joule when 10 kilovolts were applied to the capacitor. Thus, based on the tests which were conducted, none of the four sample materials will become ignited if exposed to electric discharges up to about 1 joules.

Flame Ignition Test

As will be seen in Section 6, when the procedure is presented, a flame ignition test is required when the sample material has been found to be insensitive to all the stimuli required in the sensitivity evaluation. The flame ignition test merely exposes the sample to a well characterized flame for a specified length of time. If ignition does not occur, the sample is considered to be very insensitive and no more classification testing is required. If the material is found to be sensitive in any of the previous sensitivity tests or the flame ignition test, an evaluation of the potential consequences of an ignition (e.g., mass explosion, mass fire, firespread, etc.) is necessary.

The flame ignition test is simple enough that an experimental evaluation of the test procedure was not considered necessary. In defining the tests, the following items were considered:

1. The flame will have to impinge on some powders and liquids which could be blown away if the gas stream is not gentle.



Powder samples

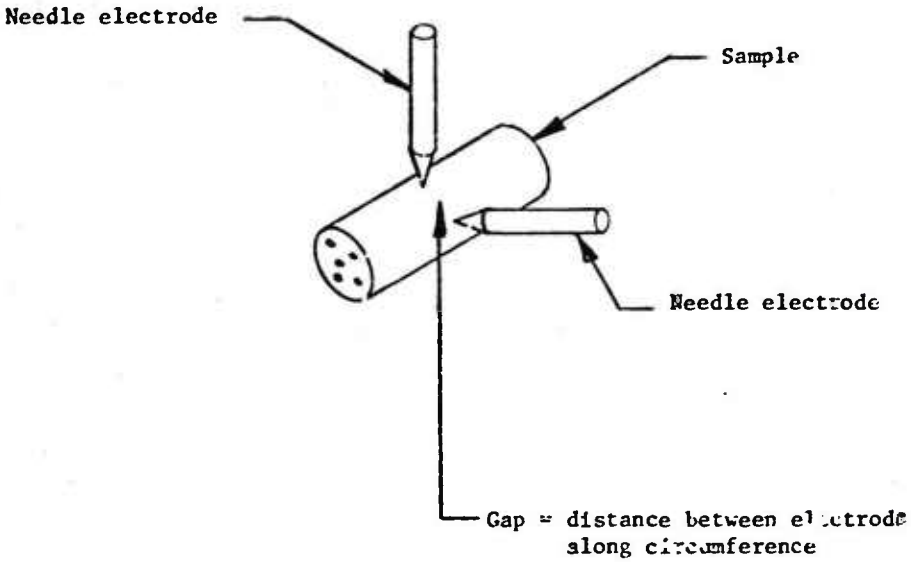


Fig 23 Sample holders for electrostatic discharge tests

Table 16

Summary of ESD ignition tests

Sample	Applied voltage (volts)	Number of trials	Remarks
M30	1000	1	No reaction
	2000	1	No reaction
	3000	1	No reaction
	4000	1	No reaction
	5000	1	No reaction
	6000	1	No reaction
	7000	1	No reaction
	8000	1	No reaction
	9000	1	No reaction
	10000	15	Burn mark observed in 4/15 trials
M1	1000	3	No reaction
	2000	1	No reaction
	3000	1	No reaction
	4000	1	No reaction
	5000	1	No reaction
	6000	1	No reaction
	7000	1	No reaction
	8000	1	No reaction
	9000	1	No reaction
	10000	22	No reaction
M26	1000	1	No reaction
	2000	3	No reaction
	10000	8	Brown spot observed in 1/8 trials
RDX	500	4	No reaction
	750	1	No reaction
	1000	1	No reaction
	2000	1	No reaction
	3000	1	No reaction
	4000	1	Powder blows away from arc
	9000	1	Powder blows away from arc
	10000	2	Powder blows away from arc

2. The flame should be inverted at, for example a 45° angle, so that it can impinge up on samples which must be held in a cup (e.g., liquids).

3. The flame should be fairly intense because this test represents the final criteria for saying that the material is very unlikely to be ignited by any means.

4. The flame should be well characterized and reproducible.

It was decided to use a standard Bunsen burner with a 9.5 mm (3/8 inch) inside diameter barrel. The burner gas should be propane and the burner should be adjusted to produce a 25 to 35 mm (1 to 1 1/4 inch) high inner cone when standing vertical in the normal position. The burner should also be adjusted to produce 960° ± 5°C at the top of the inner cone. These requirements are very close to many standard tests using a gas burner.

Once adjusted, the burner should be placed in a holder so that the flame points downward and the barrel axis is on a 45° incline to the vertical. A 5 gram sample (or a similar appropriate quantity if an inhomogeneous sample such as pellets or strands is used) should be lifted remotely into the flame so that the tip of the inner blue cone just contacts the sample. The material passes this test if no ignition occurs during the exposure. The exposure should be for a minimum of one minute.

Critical Diameter Test

The critical diameter test is a screening test in the procedure, used to help determine which effects test will be most representative of the consequence of an initiation. The critical diameter test is appropriate for materials which exist in a volume (bulk) in the process, rather than in a layer. The test is designed to answer the question, "if a detonation already exists in the process (e.g., in an adjacent vessel) can the material in this process vessel propagate the detonation?---i.e., is this material detonable?"

The apparatus chosen for this test is illustrated in Figure 24 and a photograph of the apparatus used in one test is shown in Figure 25.

The booster explosive is made long in order to produce a flat detonation front at the interface between the booster and the sample. The explosive sample is put in a tube with a length to diameter ratio of at least 6 to 1. This high length to diameter ratio was found to be necessary in order to allow the "overdriven" reaction front to stabilize and still have sufficient tube length to judge whether or not the reaction is stabilized or dying inside the sample material. Naturally, the longer the tube is, the easier it is to interpret the test results. The L/D ratio of 6 was found to be a good balance, making the tube size handleable while still being able to interpret the data. The 76 critical

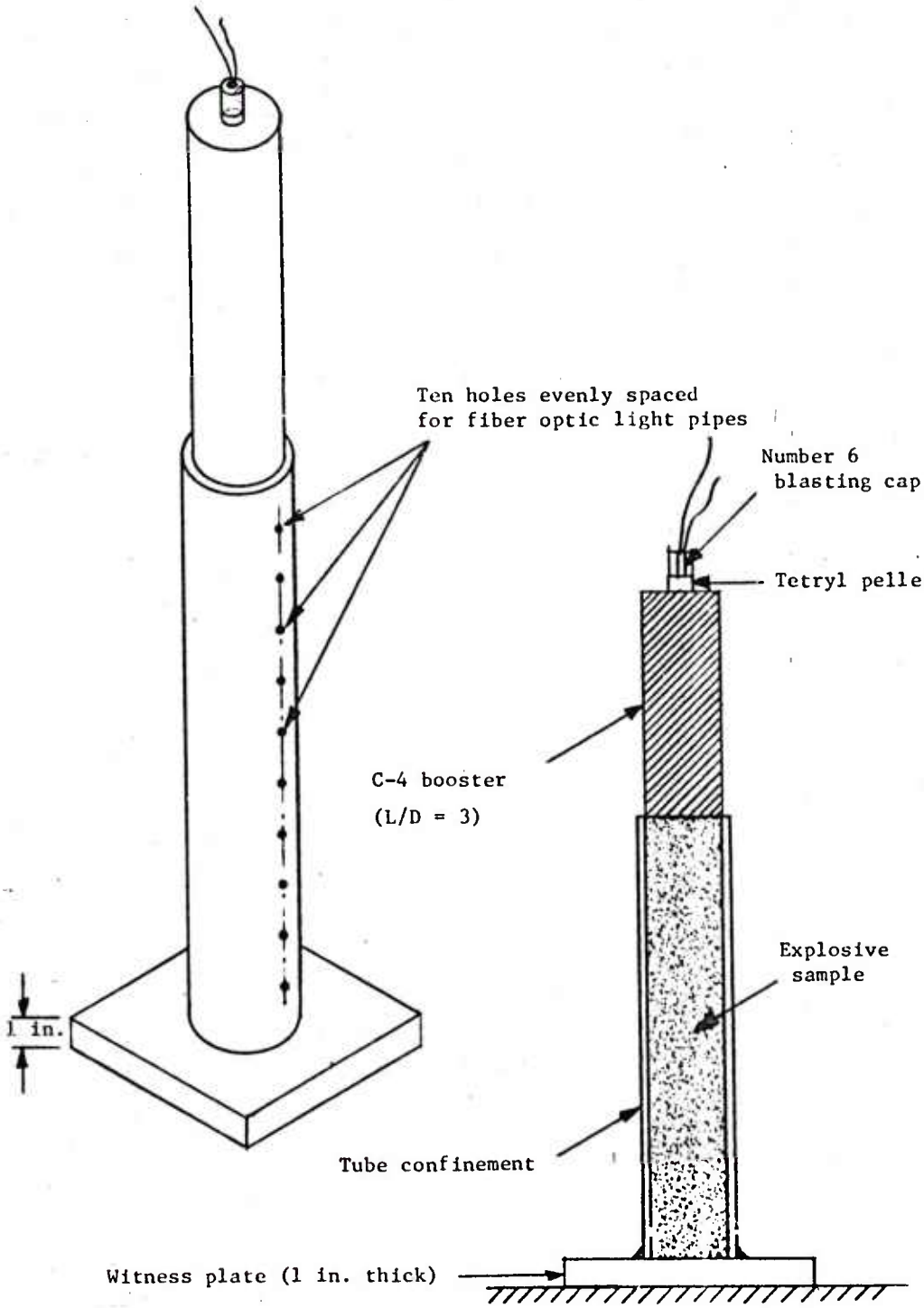


Fig 24 Critical diameter test apparatus

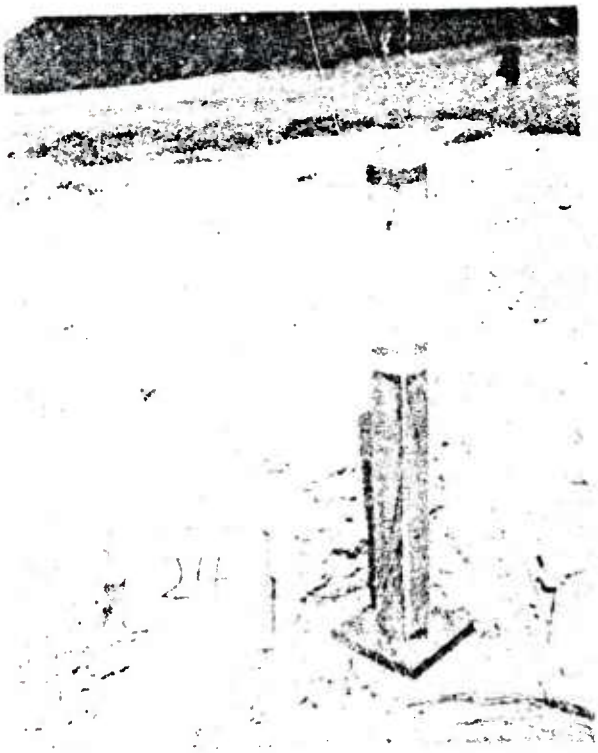


Fig 25 Critical diameter test apparatus setup in field

diameter tests which were completed are described in Table 17. The early tests concentrated on refining the test procedure, for example determining that an L/D of 4 was inadequate, requiring an L/D of 6 instead.

All four sample materials were tested in 0.318 cm (1/8 inch) walled steel tubing. To evaluate the effect of confinement, M26 was also tested in 0.635 cm (1/4 inch) walled steel tubing and cardboard tubing (effectively zero wall confinement). The 76 tests were not sufficient to accurately determine the critical diameter for all these cases, but an approximate value could be derived from the data for each case considered. Wherever possible, the Bruceton technique was used to determine the diameter corresponding to a 50 percent probability of propagation. Where insufficient data was available, other techniques were used such as grouping data to produce an approximate experimental probability curve. Figure 26 shows the sequence of tests for M26 paste in 0.318 cm (1/8 inch) wall steel tubing. Figure 27 gives the data for M26 paste in 0.635 cm (1/4 inch) walled tubing.

Figure 28 shows the sequences of tests used to determine M26 critical diameter in cardboard tubing. On the figure, Sequence 1 arrranged the available test results into the Bruceton series shown on the left side of the figure. The dotted circle is an assumed "No Go" (not actually done) which was added to Sequence 1 to produce Sequence 2.

Similarly, sequences 3 and 4 are shown on the right side of the figure. Depending on how the data was arranged, the critical diameter was found to range between 2.26 cm (0.89 in) and 3.59 cm (1.414 in).

Figure 29 shows how M26 critical diameter varies with the ratio of t/D (wall thickness to diameter ratio). The dotted line is approximately the 50 percent probability of initiation line. The four diagonal solid lines are lines of constant wall thickness. The wall thicknesses shown represent the range of practical process vessel wall thicknesses 0.08 cm to 1.27 cm (1/32 inch to 1/2 inch).

Figure 30 presents the results of tests with RDX in 0.317 cm (1/8 in) walled steel tubing. All shots down to 0.635 cm (0.25 in) diameter were positive. Since it is quite unlikely that the RDX process vessel will be that small, there was no need to carry the tests to smaller diameters. Therefore, the critical diameter for RDX slurry in 0.317 cm (1/8 in) walled tubing was found to be less 0.635 cm (0.25 in).

Figure 31 presents the results for M1 strands 0.317 cm (1/8 in) walled steel tubing. M1 strands are quite difficult to pack at a uniform density within the test volumes. Because of this, the log normal probability distribution of "Go's" was found to be quite wide. Positive reactions were found at diameters as small as 1.27 cm (0.5 in) and negative reactions were found at diameters as large as 15.2 cm (6 in). Only enough tests were completed to determine the approximate shape of the probability distribution.

Table 17

Critical diameter test results

Test	Test designation	Date	Observation of witness plate	Results	Remarks
1	CD-M30-4-.125	4/21/78	Dish plate with some shear	V[a] → 2300 m/sec STEADY GO	Tube in small fragments, should have had L/D=6 (used L/D=4)
2	CD-M30-4-.250	4/25/78	Dish with partial hole	V → 2800 m/sec STEADY GO	Tube in small fragments, should have had L/D=6 (used L/D=4)
3	CD-M30-4-.000	4/27/78	No dish plate flat and clean	V → died out NO GO	Some M30 pellets strewn around pit area
4	CD-M26-4-.25	4/27/78	Dish with hole	V → 5000 m/sec STEADY GO	Used L/D=4
5	CD-M26-4-.125	4/27/78	Dish plate with some shear	V → 4500 m/sec STEADY GO	Used L/D=4
6	CD-M26-4-000	4/28/78	Dish only	V → 4500 m/sec STEADY GO	Used L/D=4
7	CD-M1-6-.25	5/02/78	Slight dish only	V → 2300 m/sec STEADY GO	L/D=4, tube in medium size fragments
8	CD-M1-6-.125	5/02/78	Slight dish only	V → 1800 m/sec BORDER STEADY GO	Tube in small fragments, should have had L/D=6 (used L/D=4) BORDER RESULTS
9	CD-M1-6-.000	5/02/78	No dish plate flat and clean	V → died out NO GO	Used L/D=4, some M1 strands strewn around pit area
10	CD-RDX-4-000	5/04/78	Dish with hole	V → 6000 m/sec GO	Used L/D=4
11	CD-RDX-4-.125	5/04/78	Dish with hole	V → 6300 m/sec GO	Used L/D=4
12	CD-M26-2-.125	5/22/78	Dish plate with some shear	V → 8000 m/sec STEADY GO	Used L/D=4, density of M26 not at constant established but at 1.07 gm/cm^3
13	CD-M26-1-.125	5/22/78	Slight dish only	V → 8300 m/sec GO	Used L/D=4, density of M26 not at constant established but at 1.23 gm/cm^3
14	CD-C4-0.4-000	5/22/78	None used	V → 10,000 m/sec GO	Reaction front velocity way too high
15	CD-M26-2-.125	5/26/78	Dish plate with some shear	V → 6500 m/sec GO	Used L/D=4
16	CD-M26-1-.125	5/26/78	Dish with hole	V → 4000 m/sec DECREASING BORDER	Used L/D=4, border results from streak photo, witness plate indicates GO
17	CD-M26-0.5-.125	5/26/78	Dish plate with some shear	V → 6600 m/sec STEADY GO	Used L/D=4
18	CD-M26-0.25-.125	5/26/78	Slightly dish plate	V → 3500 m/sec decreasing NO GO	Use of 1 in. thick witness plate was poor choice, too thick; used L/D=4
19	CD-M26-0.25-.125	6/06/78	Dish plate with some shear	V → 3500 m/sec decreasing NO GO	Used L/D=10
20	CD-M26-0.5-.125	6/06/78	Dish plate with some shear	V → 3400 m/sec STEADY BORDER GO	Used L/D=6
21	CD-M30-1-.125	6/06/78	No dish; plate flat with some burn marks	V → 2000 m/sec decreasing NO GO	1 in. thick witness plate used, too thick; used L/D=7
22	CD-M30-2-.125	6/06/78	Dish plate, steel tube came apart in strips	V → 3300 m/sec NO GO	Material residue left on witness plate; used L/D=6

[a] V → indicates reaction front velocity approaches value shown.

Table 17 (contd)

Critical diameter test results

Test	Test designation	Date	Observation of witness plate	Results	Remarks
23	CD-M30-2.675-.125	6/27/78	Dish plate; color of plate shows high temperature	V = 1700 m/sec decreasing NO GO	Used L/D=6
24	CO-M30-4-.125	6/29/78	Dish plate	V = 1900 m/sec decreasing NO GO	Used L/D=6
25	CD-M26-.25-.125	7/07/78	No dish plate; flat with some burn marks	NO GO	Witness plate indicates NO GO. Missed scope record because nn triggering occurred. Used L/D=6.
26	CO-M26-2-000	7/07/78	Slightly dished plate	V = 4300 m/sec BORDER GO	Used L/D=6
27	CO-M26-1-000	7/07/78	No dish plate; flat with some burn marks	V = died out NO GO	Used L/D=6
28	CD-M26-1-000	7/07/78	Slight dish only	Results questionable	Missed scope record, no explanation why. No velocity could be determined. Used L/D=6.
29	CO-M26-1-.25	8/03/78	Dished plate with some shear	V = 4400 m/sec STEADY GO	Used L/D=6. Explosive losded into apparatus by increments.
30	CO-M26-1.5-000	8/03/78	Dished plate	V = 4500 m/sec; steady Slight increase GO	Used L/D=6. Explosive loaded into apparatus by increments
31	CO-M26-2-000	8/03/78	Dished plate	V = 3100 m/sec decreasing NO GO	Used L/D=6. Explosive loaded into appsratus by increments.
32	CD-M26-1-000	8/03/78	Dished plate with some shear	V = 2500 m/sec STEADY GO	Used L/D=6. Explosive loaded into appsratus by increments
33	CO-M26-1-.125	8/17/78	Dished plate with some shear	V = 4600 m/sec STEADY GO	Used L/D=6. Explosive loaded into apparatus by increments
34	CO-M26-.5-.125	8/17/78	Dished plate with some shear	V = 4100 m/sec steady Slight increase GO	Used L/D=6. Explosive loaded into spparatus by increments
35	CD-M26-.25-.125	8/17/78	No dish plate, flat with some burn marks	V = died out NO GO	Used L/D=6. Explosive loaded into apparatus by increments
36	CO-M26-.5-.125	8/23/78	Dished plate with some shear	V = 4800 m/sec GO	Used L/D=6. Explosive loaded into apparatus by increments
37	CD-M26-.5-.125	8/23/78	Dished plate with some shear	V = 3800 m/sec. Slightly decreasing border = GO	Used L/D=6. Explosive losded into apparatus by increments
38	CD-M26-.25-.125	8/23/78	Plate flat with some burn marks	V = died out NO GO	Used L/D=6. Explosive loaded into apparatus by increments
39	CO-M26-1-.125	8/23/78	Clean hole through plate	V = 5600, rising GO	Used L/D=6. Extrs M26 shot not used in Bruceton
40	CO-M26-1-.125	8/23/78	Clean hole through plate	V = 4400 to 5000 m/sec Stesdy	Used L/D=6. Exers M26 shot not used in Bruceton
41	CD-RDX-.5-.125	8/23/78	Hole almost through plate	GO by comparison of results with shot 42	Used L/D=6. Losded by Increments
42	CD-RDX-.5-.125	8/23/78	Hole almost through plate	V very stable at sbout 6000 m/sec GO	Used L/D=6. Loaded hy Incrementa
43	CO-RDX-.25-.125	8/23/78	No hole but shear	V very stahle at about 6000 m/sec GO	Used L/D=12. Loaded by Increments
44	CD-ROX-.25-.125	8/23/78	No hole but shear	V stable, rising to about 5800 m/sec GO	Used L/D=12. Loaded by increments

Table 17 (contd)
Critical diameter test results

Test	Test designation	Date	Observation of witness plate	Results	Remarks
45	CD-ROX-1-.125	8/23/78	Clean hole	V stable at about 6000 m/sec GO	Used L/D=6. Loaded by increments
46	CD-RDX-1-.125	8/23/78	Clean hole	No scope record GO based on witness plate	Used L/D=6. Loaded by increments
47	CD-M1-2.875-.125	8/24/78	Plate dished with shear	V ≈2300 m/sec STEADY GO	Used L/D=6. Density =D.55 g/cc (too high)
48	CD-M1-6-.125	8/24/78	Plate dished and ripped with shear	V ≈2700 m/sec STEADY GO	Used L/D=6. Density = 0.49 g/cc (too high)
49	CO-M26-.25-.25	9/08/78	Plate not sheared	V drops from 5000 m/sec to 3300 m/sec NO GO	Used L/D=12.
50	CD-M26-.25-.25	9/08/78	Plate slightly sheared	V stabilizes at about 3000 m/sec GO	Used L/D=12.
51	CD-M26-.25-.25	9/08/78	Plate only slightly sheared	V stabilizes at ≈ 3000 m/sec GO	L/D=12. Tube sheared at fiber optic probe holes
52	CD-M26-.5-.25	9/08/78	Some shear on witness plate	V not constant but remains above 3300 m/sec GO	L/D=6. Loaded in three increments
53	CD-M26-.5-.25	9/D8/78	Some shear on witness plate	V stabilizes at about 3500 m/sec GO	L/D=6. Loaded in three increments
54	CD-M26-.5-.25	9/08/78	Some shear on witness plate	V drops slightly but remains above 3200 m/sec GO	L/D=6. Loaded in three increments
55	CO-M26-1-.25	9/08/78	Significant witness plate shear	Significant witness plate damage indicates go GO	L/D=6. Loaded in six increments. Velocity data lost
56	CD-M26-1-.25	9/08/78	Significant witness plate shear	V stable at 4500 to 5000 m/sec GO	L/D=6. Loaded in six increments
57	CD-M26-1-.25	9/08/78	Significant witness plate shear	V stable at about 4500 to 5000 msec	L/D=5. Loaded in six increments
58	CD-M26-.5-0	9/D8/78	Apparently no witness plate damage	V drops to below 1000 m/sec ND GO	L/D=6. Loaded in three increments
59	CD-M26-1-0	9/D8/78	No witness plate damage	V drops smoothly to about 3D00 m/sec and appears stable there GO	L/D=6. Loaded in six increments
6D	CD-M26-2-0	9/08/78	Plate dished and pitted	V stable at about 4500 m/sec GO	L/D=6. Loaded in six increments
61	CO-M26-4-0	9/08/78	Plate dished, pitted and ripped	V stable at about 4500 m/sec GO	L/D=6. Loaded in six increments
62	CD-M1-1.5-.129	9/08/78	Plate dished	Velocity record lost; probably GO based on comparison of plate with test 63	L/D=6. Loaded in six increments
63	CD-M1-1.5-.125	9/08/78	Plate dished	V ≈2400 m/sec (stable) GO	L/D=6. Loaded in six increments

Table 17 (concl)

Critical diameter test results

Test	Test designation	Date	Observation of witness plate	Results	Remarks
64	CD-M1-2.875-.125	9/08/78	Plate dished	V → 2000 m/sec (stable) GO	L/D=6. Loaded in six increments
65	CO-M1-2.875-.125	9/08/78	Plate dished	V → 2000 m/sec (still dropping) BORDER	L/D=6. Loaded in six increments
66	CD-M1-6-.125	9/11/78	Plate dished and pitted	V dropping NO GO	L/D=6. Loaded in six increments
67	CO-M1-6-.125	9/11/78	Plate dished and pitted	V stabilizes at about 2000 m/sec GO	L/D=6. Loaded in six increments
68	CO-M1-.5-.125	10/02/78	Minor damage	V dropping NO GO	L/D=6.
69	CD-M1-.5-.125	10/02/78	Minor damage	V generally decreasing with large spike toward end BORDER	L/D=6.
70	CD-M1-.5-.125	10/02/78	Plate slightly dished	V stabilizes at about 2200 m/sec GO	L/D=6.
71	CD-M1-1-.125	10/02/78	Minor damage	V appears to be stabilizing at about 2000 m/sec BORDER	L/D=6.
72	CO-M1-1-.125	10/02/78	Minor damage	V dropping slowly NO GO	L/D=6.
73	CD-M1-1-.125	10/02/78	Plate slightly dished	V dropping NO GO	L/D=6.
74	CO-M1-2-.125	10/02/78	Minor damage	V dropping NO GO	L/D=6.
75	CD-M1-2-.125	10/02/78	Minor damage	V dropping NO GO	L/D=6.
76	CO-M1-2-.125	10/02/78	Plate slightly dished	V seems to stabilize at about 2000 m/sec GO	L/D=6.

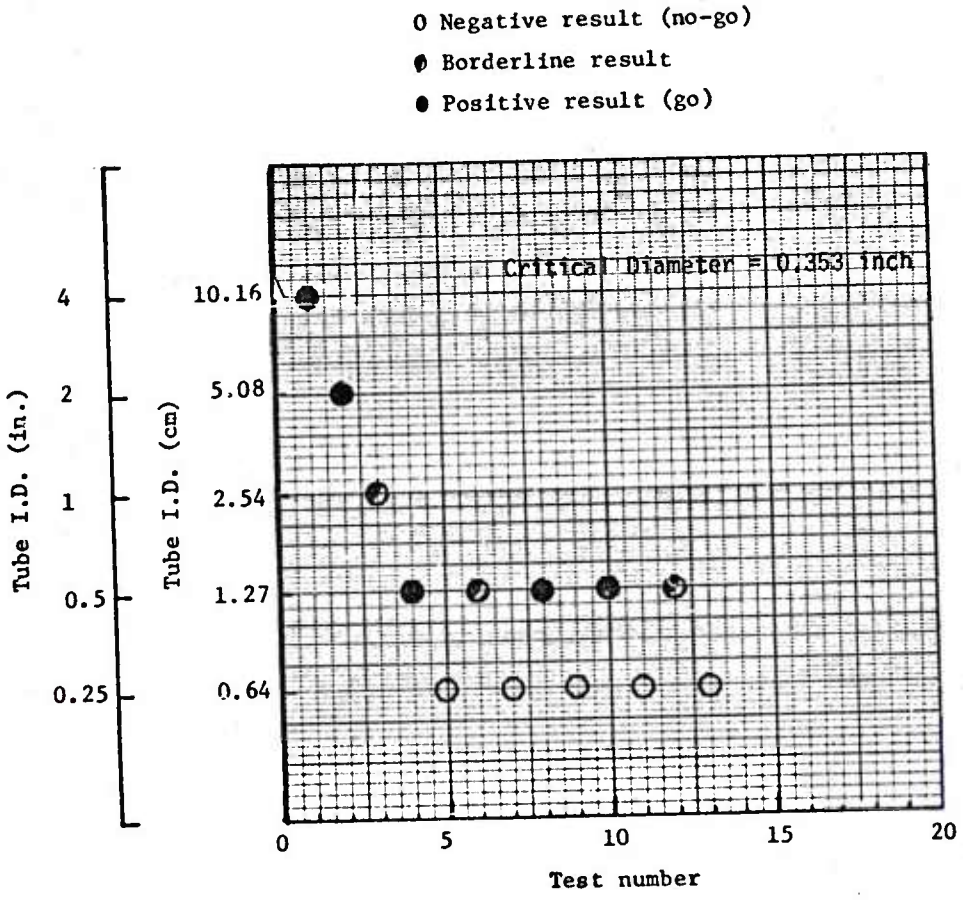


Fig 26 Critical diameter determination for M26 paste in 0.318 cm (1/8 in.) wall tubing

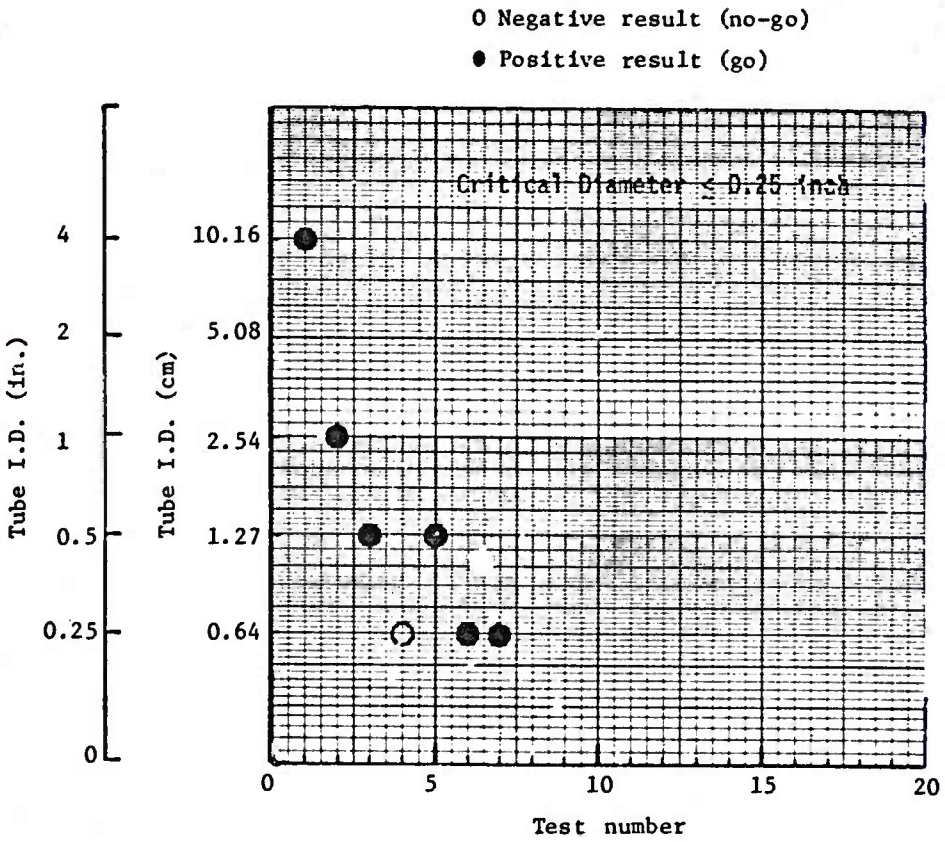


Fig 27 Critical diameter determination for M26 paste in 0.635 cm (1/4 in.) wall tubing

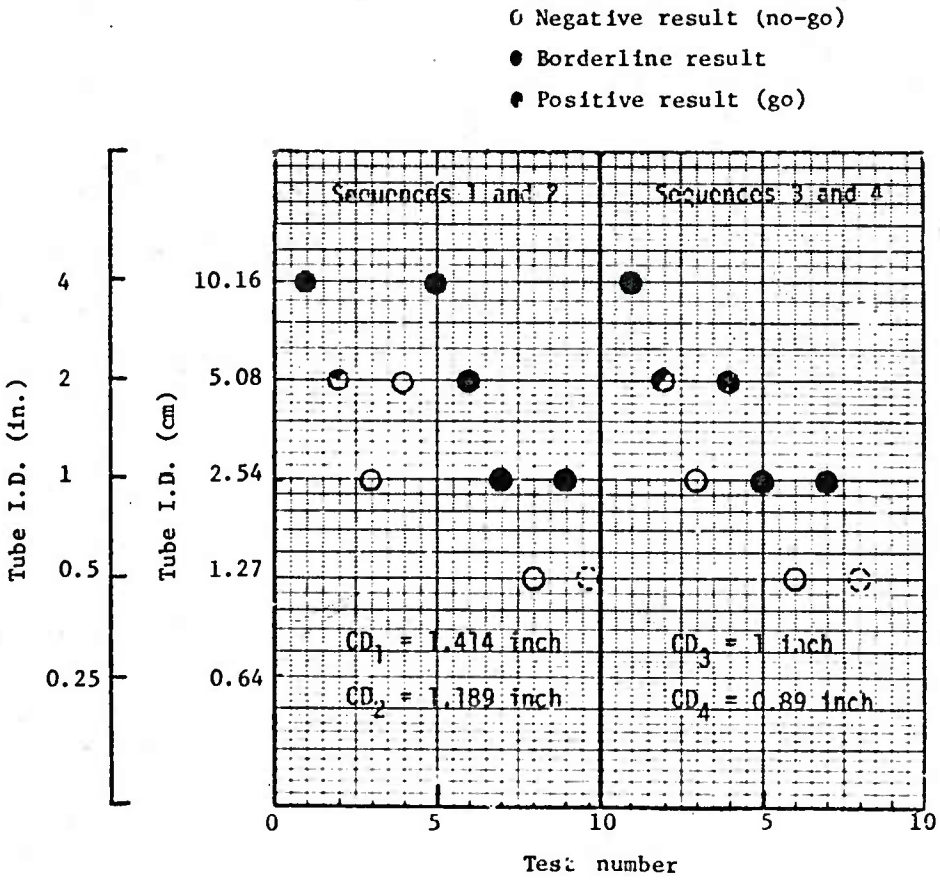


Fig 28 Critical diameter determination for M26 paste in cardboard tubing

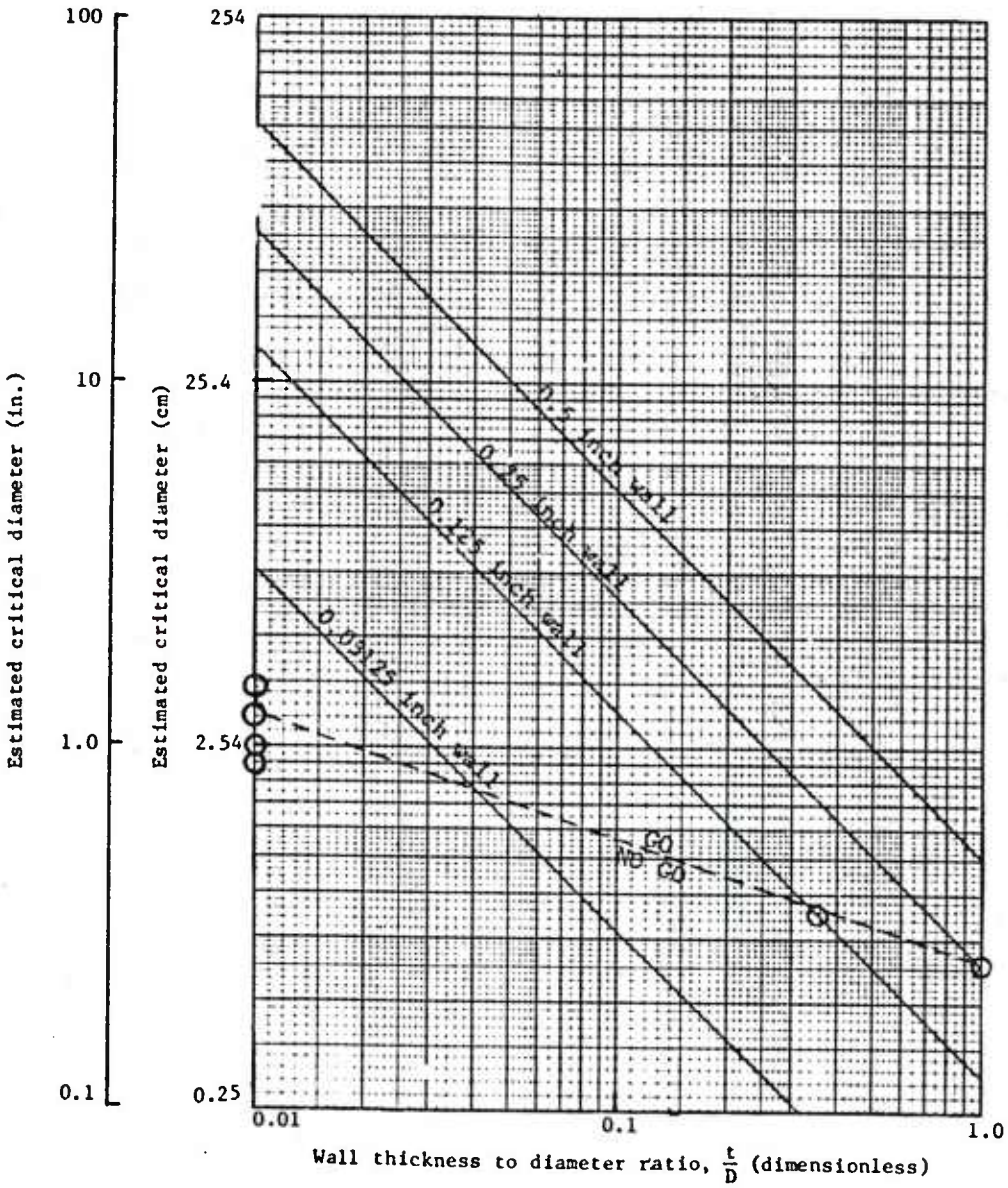


Fig 29 Relation of critical diameter to tube wall thickness for M26 paste

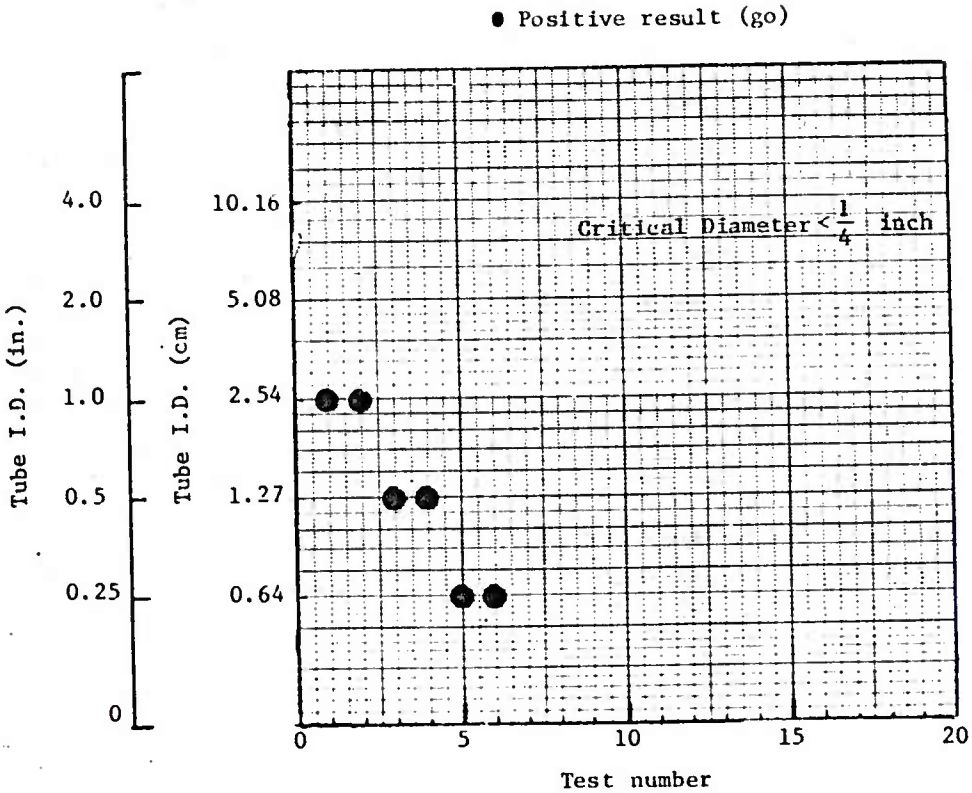


Fig 30 Critical diameter determination for RDX slurry in 0.318 cm (1/8 in.) wall tubing

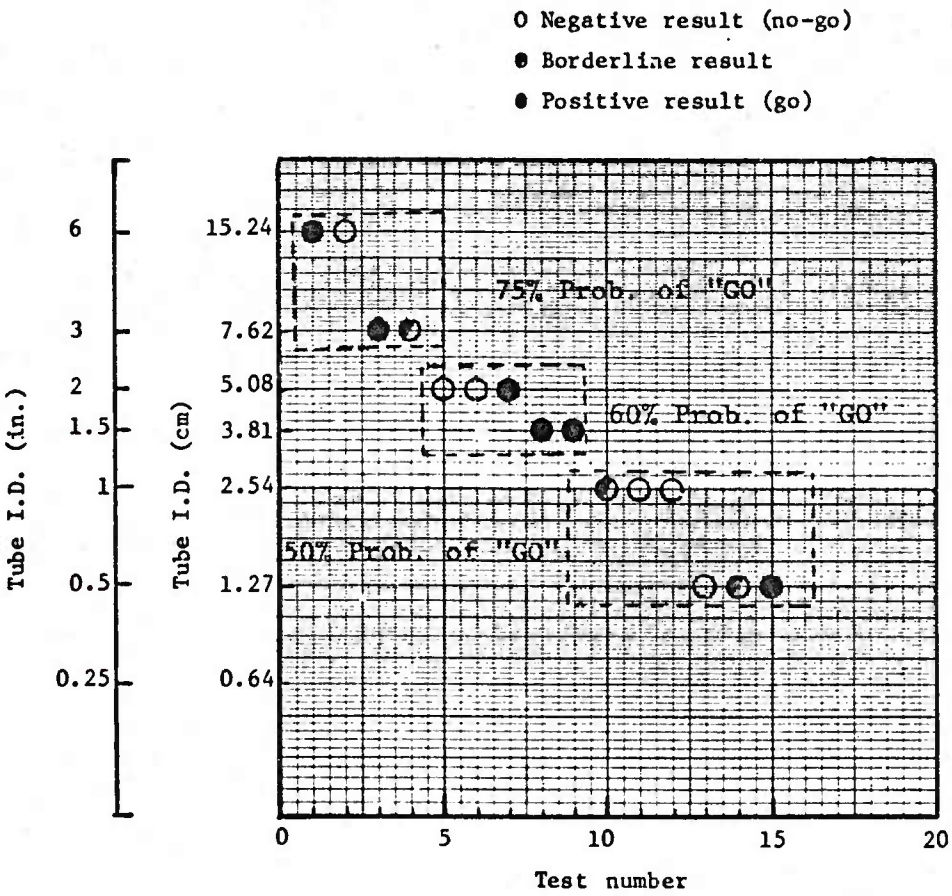


Fig 31 Critical diameter determination for M1 strands in 0.318 cm (1/8 in.) wall tubing

From the data in Figure 31, it appears that the critical diameter is about 1.9 cm (0.75 in.), although depending on how the data are reduced, critical diameter can be anywhere in the range from 1.4 cm (0.56 in.) to 6 cm (2.38 in.).

Each critical diameter test had to be evaluated to determine whether or not stable detonation was established in the sample material. Generally, the C4 explosive booster will provide an initiating detonation front much stronger than the stable detonation condition in the sample (the sample will be overboostered). The first half of the tube length will involve the detonation front stabilizing from the overboostered state to the stable reaction front velocity for the sample. If the reaction front stabilizes in the second half of the tube length at a nearly constant (some oscillations are expected) supersonic velocity, on the order of 2000 to 8000 m/s (7000 to 26,000 ft/s), a stable detonation has been established in the sample and the tube diameter is above the critical diameter. If the reaction front velocity continues to attenuate in the second half of the tube, a stable detonation has not been established and the tube diameter is below the critical diameter.

In Figure 32, three typical plots of reaction front velocity versus distance derived from the experimental data are shown. The top curve shows the velocity stabilizing at about 2250 m/s. The middle curve shows the velocity dropping, but it appears that it could have stabilized had the tube been a little longer. The bottom curve shows the reaction front velocity dropping steadily. The top curve was considered to be a "Go". The middle curve was a "No Go" since even if the velocity had stabilized it would have been well below 2000 m/s. This was a somewhat borderline situation. The third test was clearly a "No Go". In many other cases, "No Go" reactions were even more obvious with the velocity probe signals weekening in intensity with the reaction and in some instances accompanied by unreacted material left on the witness plate.

Three examples of data taken using fiber optic "light pipes" as shown in Figures 24 and 25 are given in Figure 33. The light from the reaction front was transmitted to a photo cell using the fiber optic "light pipes". Each time the reaction front passed a probe, the photo cell voltage spiked. The oscilloscope records in Figure 33 represent time on the horizontal axis and voltage (light intensity) on the vertical axis. Average velocity between two probes was calculated by dividing the distance between the probes at the steel tube by the time between signals on the oscilloscope record. The time between signals was measured from spike to spike when the signals were sharp or between corresponding locations at which the signal rose sharply when the peaks were not as narrow.

Another useful qualitative indication of a detonation is shearing of a hole through the 2.54 cm (1 in.) thick steel witness plate. In order for a hole to be sheared into the plate, the reaction front velocity must be greater than the speed of sound in the plate (about 5945 m/s [19,500 ft/s]) at the end of the tube. Such a high velocity would have to be

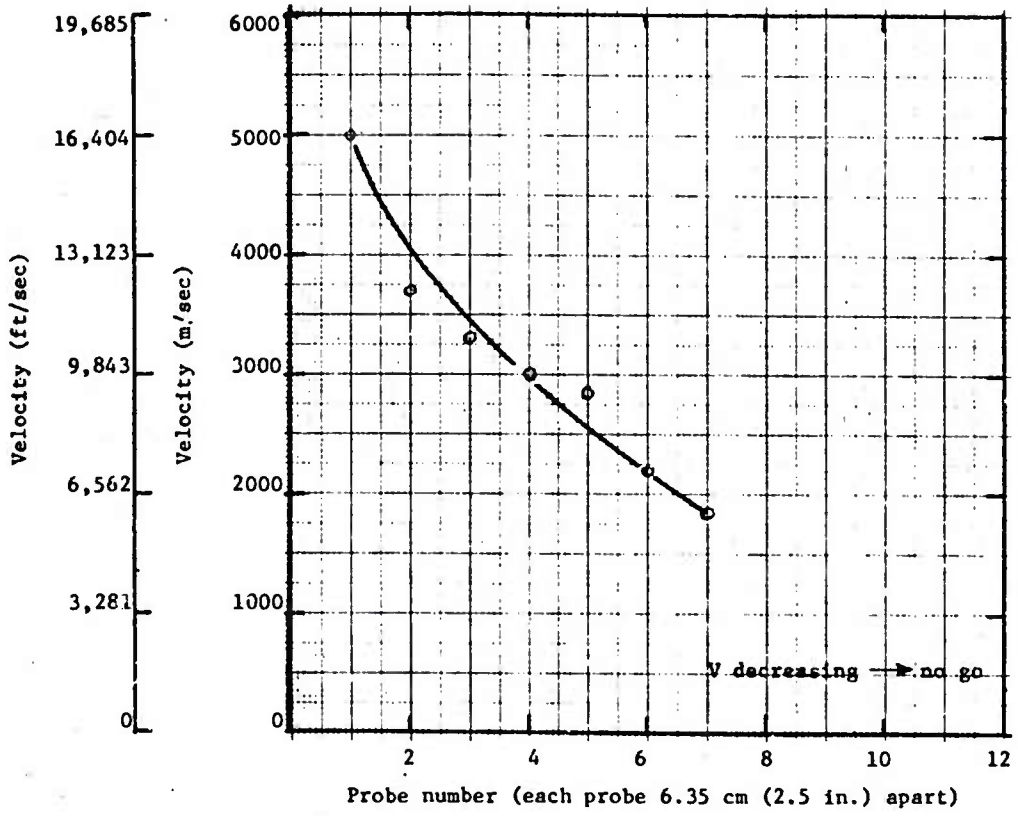


Fig 32(a) Critical diameter test 24, velocity profile

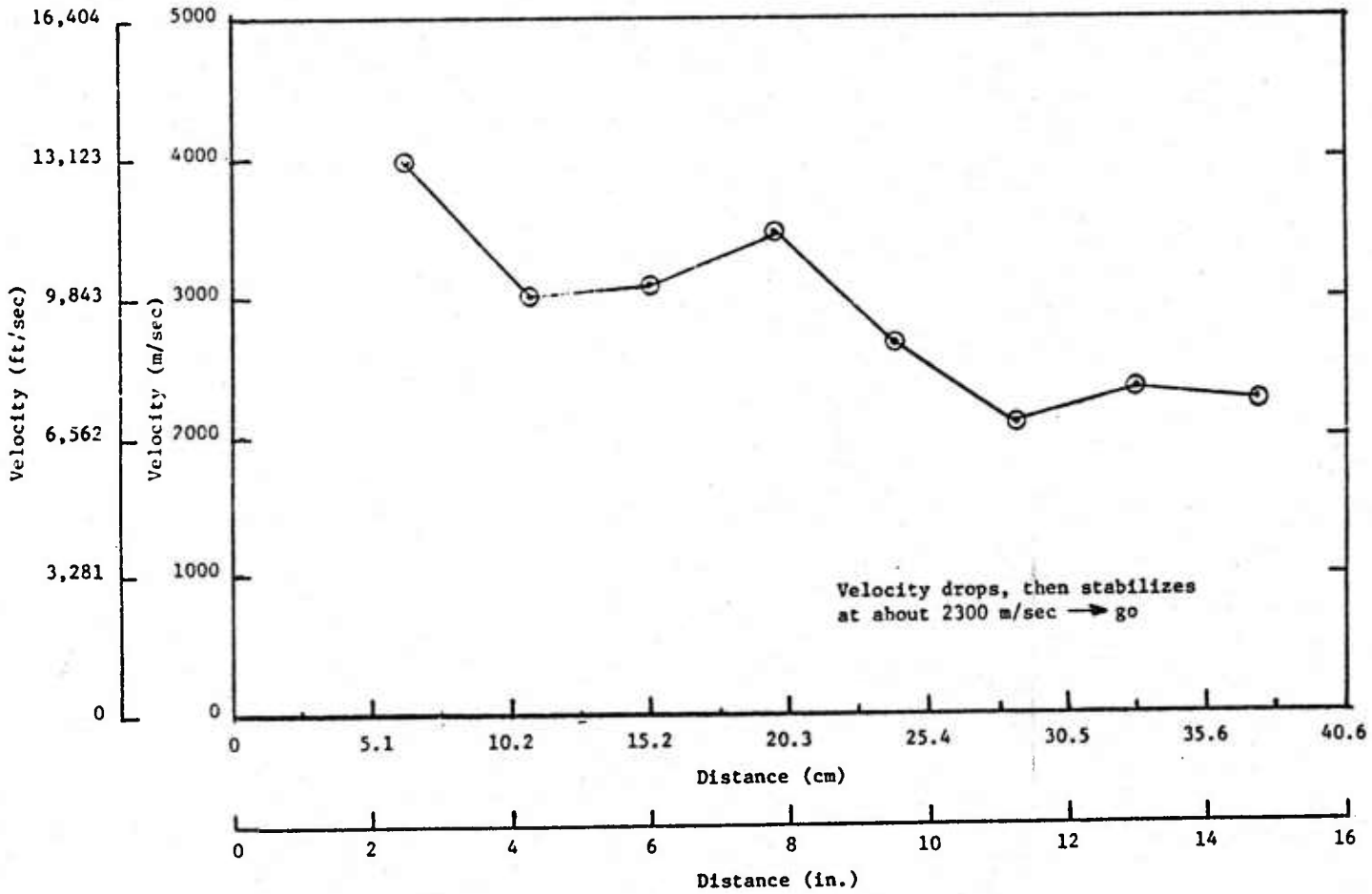


Fig 32(b) Critical diameter test 47, velocity profile

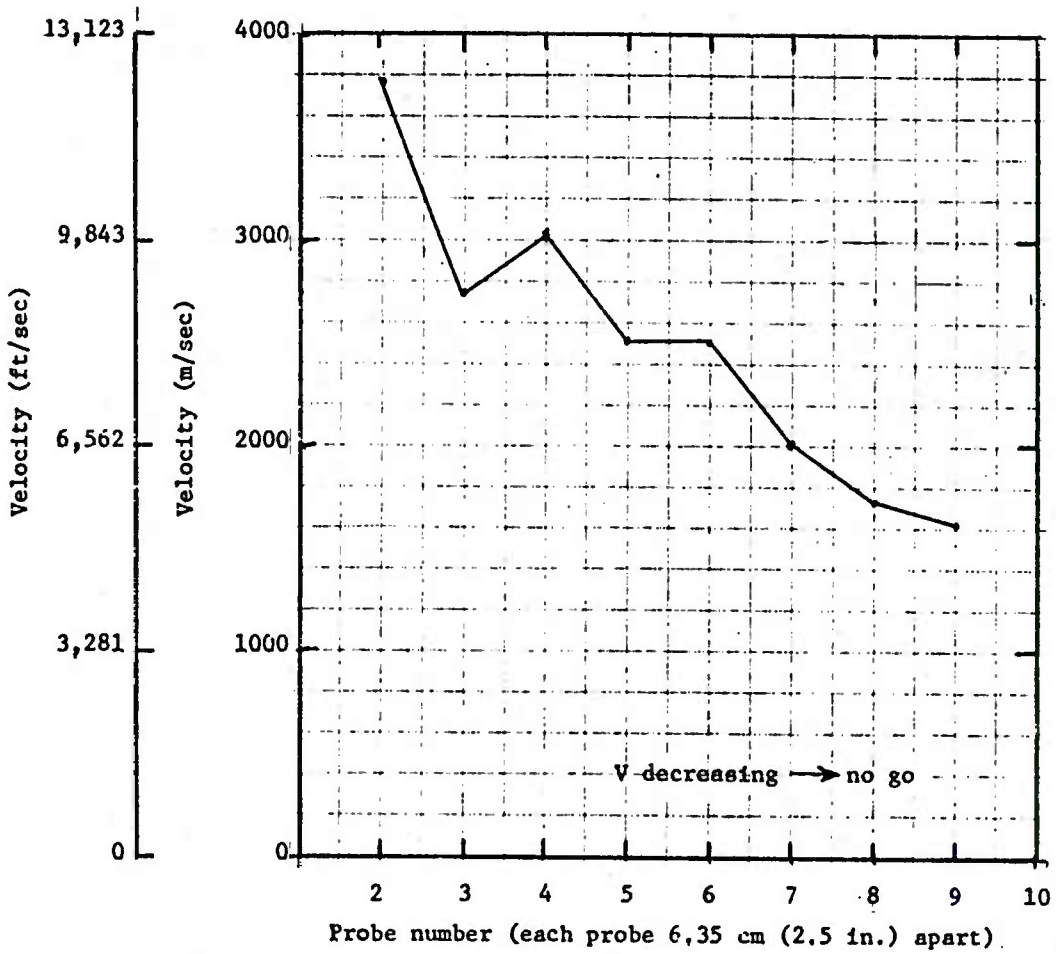


Fig 32(c) Critical diameter test 75, velocity profile

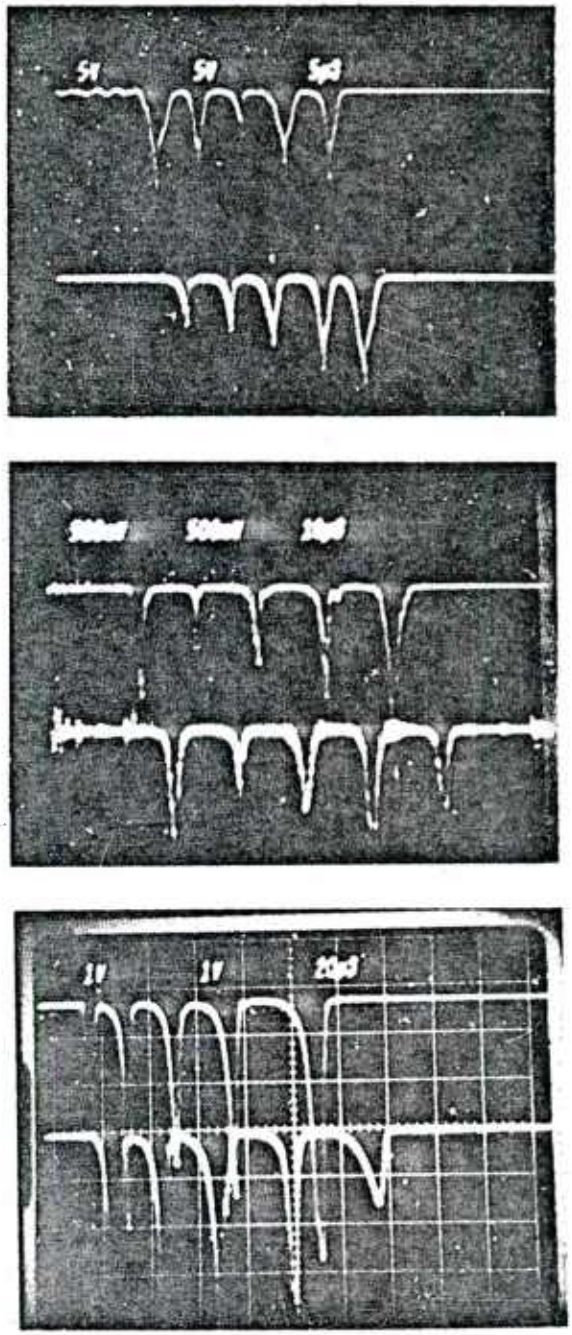


Fig 33 Typical oscilloscope traces from fiber optic velocity probe

associated with a detonation, therefore a hole sheared through the plate is a good indicator of a positive result. The absence of a hole sheared through the plate does not necessarily indicate a negative reaction since the detonation velocity could just be too low. However, absence of a hole generally does correspond to non-detonation or a weaker detonation than is typical for most condensed explosives.

In refining the critical diameter test procedure, two more issues had to be resolved. First, since the test uses a circular cylindrical tube, the derived critical diameter is only directly extrapolateable to process vessels which are circular cylinders. In project SOPHY (Ref. 16) equations were derived which can be used to compute an equivalent vessel diameter for vessel cross-sections which are not circular (e.g., triangles, rectangles, etc.). These relations have been incorporated into the critical diameter test procedure. Second, consideration had to be given to the situation where, perhaps due to facility limitations, the testing cannot be done at large enough diameters to obtain positive results (Go's) and the actual process vessel is larger than the allowable facility size limitation. In such cases, the actual (full scale) vessel may be able to propagate a detonation whereas the largest tests show only negative results. Several approaches can be used to resolve this problem. Among these are the following:

1. Analytical Estimation of an Energy TNT Equivalency

   If the chemical composition of the reactants is known, the energy released, assuming the mixture detonates, can be predicted analytically by a number of methods. Using thermochemical tables and simple assumptions as to what the products will be, a simple calculation can be done to obtain an approximation of the energy released. Alternatively, a computer code such as TIGER (Ref. 17) or EQUAL (Ref. 18) can be used to obtain a more accurate prediction. The energy release predicted for the process chemical can be divided by the value predicted for TNT to estimate a TNT equivalency dependent purely upon the energy release. This approach is conservative in that it assumes that detonation will occur while detonation may not actually be possible. The approach may not be acceptable to the user in that it requires that somewhat complex calculations must be conducted.

2. Dictate Classification by Policy

   If the critical diameter cannot be determined by test, a second approach is to admit the lack of knowledge and base the classification decision purely on policy. From the standpoint of safety conservatism, the logical decision would be to classify all such materials as Class 1.1A (mass explosion hazard). From the standpoint of economic conservatism, the logical decision would be to choose class 1.4 (minor hazard) and design the process plant accordingly.

3. Base Decision on Effects Test Results

The third approach is the one which has been selected for the hazard classification procedure. This approach is to base the decision on the results of the effects testing. The mass explosion test is done first. If the TNT equivalency is found to be greater than or equal to 10 percent, the material is classified as 1.1A (mass explosion hazard). If the equivalency is less than 10 percent, the mass fire test is done to determine the classification.

Tube Transition Test

The purpose of this test is to determine the process vessel length required for a burning reaction (flame) to transition into a detonation in the process material being evaluated. The test is for materials in a volume (bulk) as opposed to materials in a layer configuration. The "critical height" test has been a somewhat standardized technique for obtaining this information. A short description of this test (taken from Ref. 19) is presented in Figure 34. This test appears to be a realistic simulation of the transition from a submerged flame to a detonation in process vessels such as hoppers or other bulk material storage containers.

When reviewing this test, several problems were noted. First, black seamless schedule 40 pipe may or may not be representative of the actual process vessel wall being evaluated. This type of problem turned out to be unavoidable in the final test configuration chosen for this hazards classification procedure. This will be discussed later. Second, the height to which material is filled in the pipe and the overall pipe length are variables which will influence the results. In order to minimize the number of tests required, these variables should be fixed in some way that is representative of the actual process vessel being evaluated, e.g., fix the ratio of material height to tube height. Third, if the test sample material is in a loosely packed form, as many process materials are, and the transition to detonation or explosion is relatively slow, the sample material will be blown out of the top of the tube making the interpretation of results somewhat difficult. Fourth, and most important, the criteria for a positive reaction in the critical height test is an "explosive reaction" of any type, not necessarily a detonation. From the standpoint of hazard, an explosive reaction of any type is the concern. It really doesn't matter as much whether or not a detonation is achieved. However, from the standpoint of minimizing the number of variables which must be considered as well as interpretation of results, a detonation is a much cleaner criteria. A nondetonating explosion of the test pipe is primarily a problem of pipe structural failure. The response of the pipe wall (which in the test may not be representative of the actual process vessel) to the pressure buildup inside is the primary phenomena being tested. In addition, the length of empty pipe above the top of the sample in the test will add strength to hold the pipe

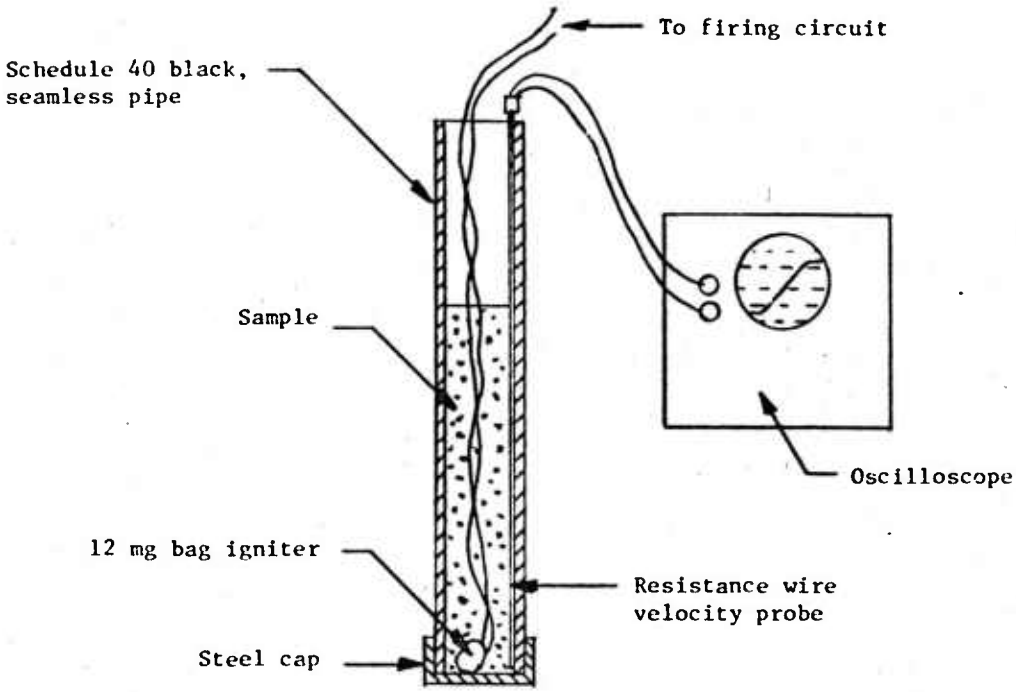


Fig 34 Critical height to explosion test setup (from reference 20)

together during a pressure (nondetonating) explosion. Because of the increased number of variables which must be considered and increased uncertainties in interpreting the results with "explosion" as the criteria, it was decided to use buildup to detonation as the criteria for transition in the hazard classification procedure. With detonation as the criteria, variables such as pipe length and void space length should only have a minor influence, if any. In addition, to avoid blowing out the sample through the open top of the apparatus, it was decided to close off the test container. If this affects the results in a test, it will shorten the length for buildup to detonation and yield a more conservative answer.

The "tube transition" tests conducted under this project are described in Table 18. Three types of apparatus are referred to in the table. These reflect different concepts which were evaluated during the program. The three test apparatus types are described in Figure 35. These represent an evolution forced by problems encountered at each step. The type 1 apparatus is similar to the "critical height" test shown in Figure 34 except the tube is completely full of sample at the actual process bulk density, it is capped at both ends (witness plate added), and the wall thickness and material is that used in the actual process vessel. Tests are begun at a diameter approximately 20 percent longer than the critical diameter and the diameter is increased to develop a curve of critical length for transition versus tube inside diameter. The critical length should generally increase as the diameter is increased, since a single sized initiator is used regardless of diameter. In an attempt to further idealize the test and identify a single characteristic critical length, the Type 2 apparatus was tried. This configuration was the same as Type 1 except the initiation source was maintained at a constant energy per unit area with a constant density of squib initiators. By maintaining a constant initiation energy per unit area, the critical length should decrease asymptotically to a constant (large diameter) critical length value. Unfortunately, both the Type 1 and 2 configurations had the same very important weakness. Because the tube walls were generally quite weak compared to the internal pressure buildup, many times the tubes exploded from the internal pressure well before a detonation could be established. Alternatively, the cap or plate at the initiation end would be blown off or punched through. Based on the Type 1 and 2 test results, it was clear that the vessel would have to be quite strong in order to allow a detonation to develop in many sample materials. The Type 3 configuration eventually evolved. Only three tests were conducted in this apparatus (one with RDX, one with M26 and one with MJ). This was not enough to unquestionably validate the test, however, the results were quite promising and the procedure for the tube transition test was based on this configuration. It is suggested that a more extensive experimental evaluation of this test configuration be conducted prior to imposing it as a hazards classification test requirement.

Table 18

Tube transition test results

Test	Test designation	Date	Observation of witness plate	Results	Remarks
1	CL-M26-1-.125 Type 1	7/07/78	Slightly dished plate	No velocity available; visual observation of apparatus indicates critical length at 41.9 cm.	Problem in oscilloscope triggering and recording; bad scope record obtained, used light pipes and scope.
2	CL-M26-2-.125 Type 1	7/07/78	Dished with hole through	No velocity available, visual observation of apparatus indicates critical length at 30.5 cm.	Problem in oscilloscope triggering and recording; bad scope record obtained, used light pipes and scope.
3	CL-M26-.5-.125 Type 1	7/25/78	Slightly dished plate	No velocity available, visual observation of apparatus indicates critical length at 29.5 cm.	Problem in oscilloscope triggering and recording, bad scope record obtained, used continuous resistance probe.
4	CL-M26-1-.125 Type 1	7/20/78	Dished with hole through	V=4200 m/sec, calculated critical length 17.3 cm, observed critical length 22.5 cm.	Good record, used continuous resistance probe.
5	CL-M26-4-.125 Type 1	7/20/78	Slightly dished plate	No velocity available, visual observation, used a 45.7 cm long tube, for 20.3 cm the tube held then split and twisted for remaining length, no critical length could be determined.	Bad scope record obtained, used continuous resistance probe. See Note 1.
6	CL-M26-2.875-.125 Type 1	7/20/78	Slightly dished plate	No velocity available; visual observation, used a 45.7 cm long tube; it split the entire length; no critical length could be determined.	Problem with oscilloscope triggering, bad scope record obtained, used continuous resistance probe. See Note 1
7	CL-M26-2-.125 Type 1	7/25/78	Dished with hole through	No velocity available, visual observation of apparatus indicates critical length at 30.8 cm.	Problem in oscilloscope triggering, bad scope record obtained, used continuous resistance probe. See Note 1.
8	CL-M26-2.875-.125 Type 1	8/01/78	Slightly dished plate	No velocity available; visual observation; some unburned material on witness plate indicating no detonation, no critical length could be determined.	Bad scope record obtained, used continuous resistance probe, cover and witness plates backed by earth. See Note 1.
9	CL-M26-4-.125 Type 1	8/01/78	Slightly dished plate	No velocity available, visual observation, some unburned material thrown over field indicating no detonation; no critical length could be determined.	Bad scope record obtained; used continuous resistance probe, cover and witness plates backed by earth, both thrown from pit. See Note 1.
10	CL-M26-.5-.125 Type 2	8/01/78	Dished with some shear		

Table 18 (contd)

Tube transition test results

Test	Test designation	Date	Observation of witness plate	Results	Remarks
11	CL-M26-1-.125 Type 2	8/01/78	Dished with hole through	No velocity available, visual obaervation of apparatus indicates critical length at 22.9 cm.	Used one S-65 squib; bad scope record obtained; used continuous resistance probe. See Note 1.
12	CL-M26-2-.125 Type 2	8/02/78		No velocity available, visual observation, some material around, no critical length could be determined.	Used five S-65 squibs; bad scope record obtained; used continuous resistance probe See Notes 1 and 2.
13	CL-M26-2.875-.125 Type 2	8/02/78	Dished plate	No velocity available, visual observation, some unburned material around, no critical length could be determined.	Used nine S-65 squibs; bad scope record obtained; used continuous resistance probe. See Notes 1 and 2.
14	CL-M26-4-.125 Type 2	[illegible]78	Slightly dished plate	No velocity available, visual observation, some unburned material around; no critical length could be determined.	Used 13 S-65 squibs; bad scope record obtained, uaed continuous resistance probe. See Notes 1 and 2.
15	CL-M1-.5-S40 Type 2	10/03/78	Plate still attached to pipe	Detonation did not develop within the 61 cm long pipe.	Initiation by a 2.54 cm deep black powder layer with one squib per each square inch.
16	CL-M1-1-S40 Type 2	10/03/78	Negligible damage	Detonation did not develop within the 61 cm long pipe.	Initiation by a 2.54 cm deep black powder layer with one squib per each square inch
17	CL-M1-2-S40 Type 2	10/03/78	Negligible damage	Detonation did not develop within the 61 cm long pipe	Initiation by a 2.54 cm deep black powder layer with one squib per each square inch
18	CL-M1-4-S40 Type 2	10/03/78	Plate still attached to pipe	Detonation did not develop within the 61 cm long pipe	Initiation by a 2.54 cm deep black powder layer with one squib per each square inch
19	CL-M26-.5-S40 Type 2	10/03/78	Negligible damage	Critical length is about 47 cm 0[a] 2540 m/s	Initiation by a 2.54 cm deep black powder layer with one squib per each square inch
20	CL-M26-1-S40 Type 2	10/03/78	Plate dished	Critical length is about 34.5 cm. 0[a] 3900 m/s	Initiation oy a 2.54 cm deep black powder layer with one squib per each square inch
21	CL-M26-2-S40 Type 2	10/03/78	Plate shattered into numerous pieces	Critical length is about 44.7 cm 0 = 7600 m/s	Initiation by a 2.54 cm deep black powder layer with one squib per each square inch
22	CL-M26-4-S40 Type 2	10/03/78	Plate slightly dished	Apparently did not establish a detonation	Initiation by a 2.54 cm deep black powder layer with one squib per each square inch

[a]Established detonation velocity

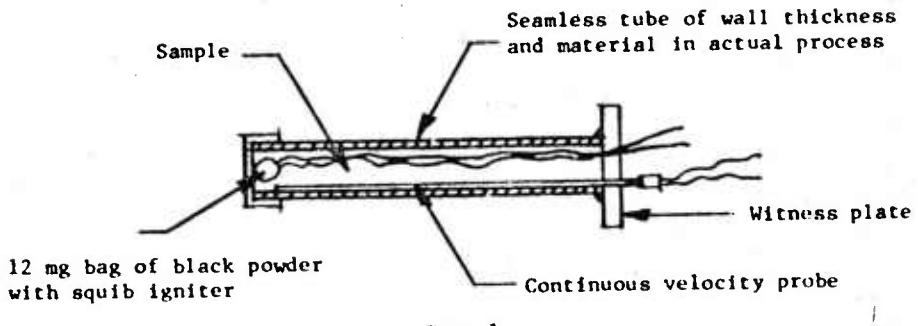
Table 18 (concl)

Tube transition test results

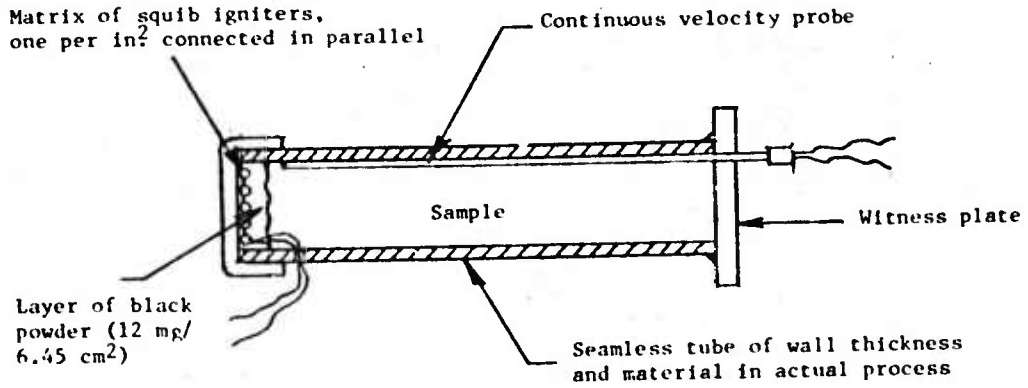
Test	Test designation	Date	Observation of witness plate	Results	Remarks
23	CL-RDX-.5-S40 Type 2	10/04/78	Clean hole through plate	Critical length is about 24.9 cm. D ~3550 m/s	Initiation by a 2.54 cm deep black powder layer with one squib per each square inch.
24	CL-RDX-1-S40 Type 2	10/04/78	Clean hole through plate	Critical length about 1.27 cm. Detonation velocity about 1400 m/s	Initiation by a 2.54 cm deep black powder layer with one squib per each square inch.
25	CL-RDX-2-S40 Type 2	10/04/78	Plate still attached to long banana peels of pipe	Data not useable; apparently did not detonate, pipe banana peeled along entire length.	Initiation by a 2.54 cm deep black powder layer with one squib per each square inch.
26	CL-RDX-4-S40 Type 2	10/04/78	Plate broken into several pieces	Critical length ~ 5.1 cm Detonation velocity 6277 m/s	Initiation by a 2.54 cm deep black powder layer with one squib per each square inch.
27	CL-M26-2.5-S160 Type 3	11/78	Missing	Critical length is approximately 20.3 cm based on fragments and 9.1 cm based on continuous velocity probe.	None
28	CL-RDX-2.5-S160 Type 3	11/78	Circular slug found	Almost instantaneous transition to detonation.	None
29	CL-M1-2.5-S160 Type 3	11/78	Minor	Detonation did not develop within the 121.9 cm pipe length.	None

Notes:

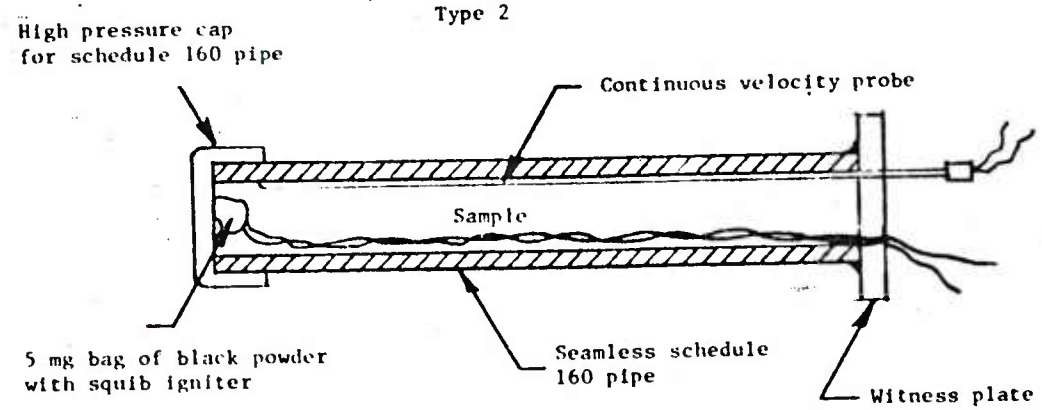
1. Pressure vesrel explosion - tube wall and/or cover plate failed before detonation could be established
2. Type 1 series uses one initiating S-65 squib with 12 gm of black powder in cloth bag.
3. Type 2 series uses one initiating S-65 squib per square inch of cross section with 2.54 cm thick layer of black powder at a bulk density of 0.96 gm/cm^3.



Type 1



Type 2



Type 3

Fig 35 Tube transition test types

Type 3 Critical Length Test Results

The three Type 3 tests which were completed are described in Table 19. Tests were conducted using M26 paste, RDX slurry and M1 strands. These tests were done primarily to evalute the test method rather than to determine the critical lengths. To reliably determine the critical length for any material, several repeats should be done at each condition and tests should be done at several diameters. The shortest critical length result as extrapolated to the full scale should be chosen.

Table 19

Type 3 tube transition

Tests completed

Designation	Material	Sample weight
CL (Type 3) 1	M26	2.31 kg (5.09 lb)
CL (Type 3) 2	RDX	3.11 kg (6.84 lb)
CL (Type 3) 3	M1	1.25 kg (2.76 lb)

- Loaded in 20 increments to obtain relatively uniform density
- Tested with pipe horizontal in arena

The setup for the RDX slurry test is shown in Figure 36. The top photograph shows the initiating end of the pipe after (1) the witness plate was welded to the far end of the pipe, (2) the continuous velocity probe and squib leads were positioned along the inside pipe walls, (3) the sample was loaded in increments to obtain a fairly uniform density, and (4) the 5 gram black powder bag with S65 squib were connected to the squib leads and placed at the end of the pipe. Next the pipe cap was screwed on and the pipe positioned in the field as shown in the lower photograph. The horizontal orientation is used to catch the fragment remains in an arena.

The fragment remains from the three tests are shown in Figure 37. The continuous velocity probe results for these tests were not quite as clear as those obtained in many of the prior tests, but corresponded essentially to the fragment results. The RDX (from fragments and scope record) initiates detonation almost instanteously. Thus, for RDX slurry the critical length is negligible. For M26, the fragment remains indicate that the critical length is approximately 20.3 cm (8 in). The scope record indicated that the critical length is 9.1 cm (3.6 in). The prior critical length test results also gave a longer critical length based on the fragments than based on the continuous velocity probe scope record. The smaller value is more conservative and should be used. Both the fragments and the scope record indicate that the M1 strands will not develop a detonation within 1.22 m (4 ft). Therefore, the critical length for M1 is greater than 1.22 m and perhaps M1 strands cannot develop a detonation in the 6.35 cm (2.5 in) ID tube used.

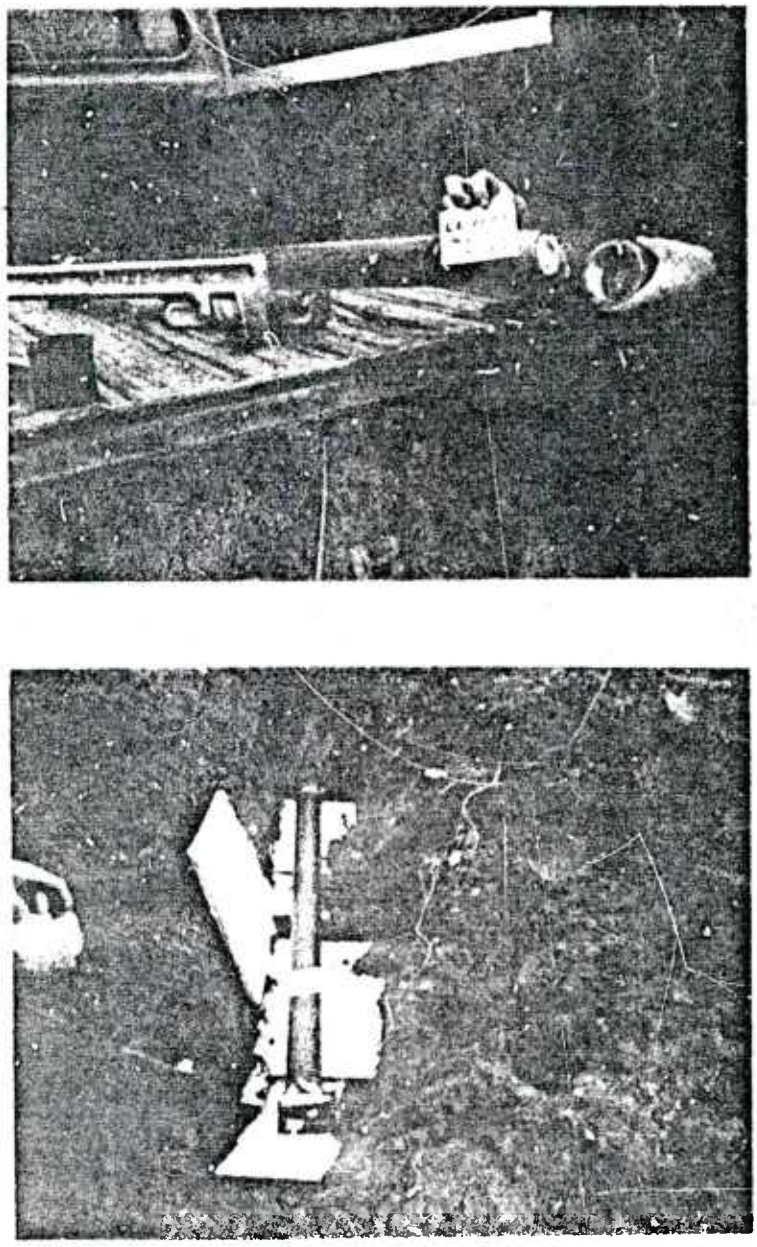
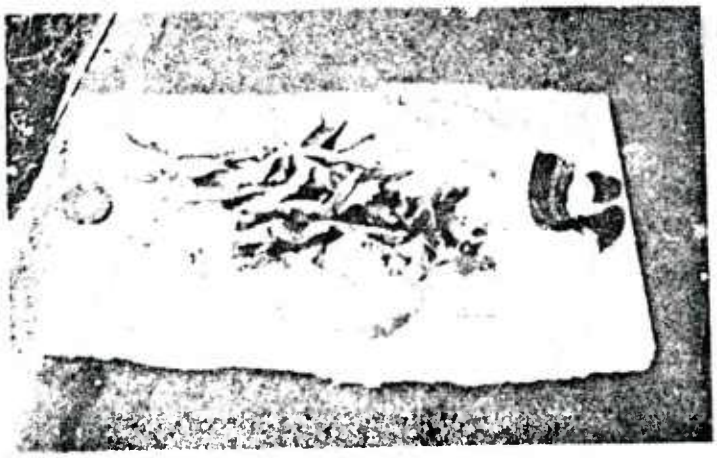
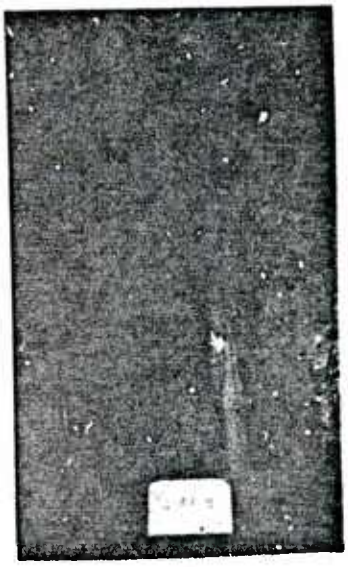


Fig 36 Type 3 critical length test setup



(a) RDX slurry

(b) M26 paste



(c) M1 strands

Fig 37 Type 3 critical length test fragment remains

Critical Length Instrumentation

As was mentioned earlier, the critical length can be determined by two methods: from the fragment remains or by measuring the reaction front velocity. The technique used to estimate critical length from the fragment remains is illustrated in Figure 38. To measure the reaction front velocity, continuous resistance velocity probes were used, however, rather than using thin soft aluminum tubing for the probe outer casing as is typically done, thin stainless steel tubing was used. This made the probe infinitely more durable. It would have been quite difficult to load the test vessels without crushing the aluminum tubing. The steel tubing was quite rugged while of negligible strength in the detonation environment.

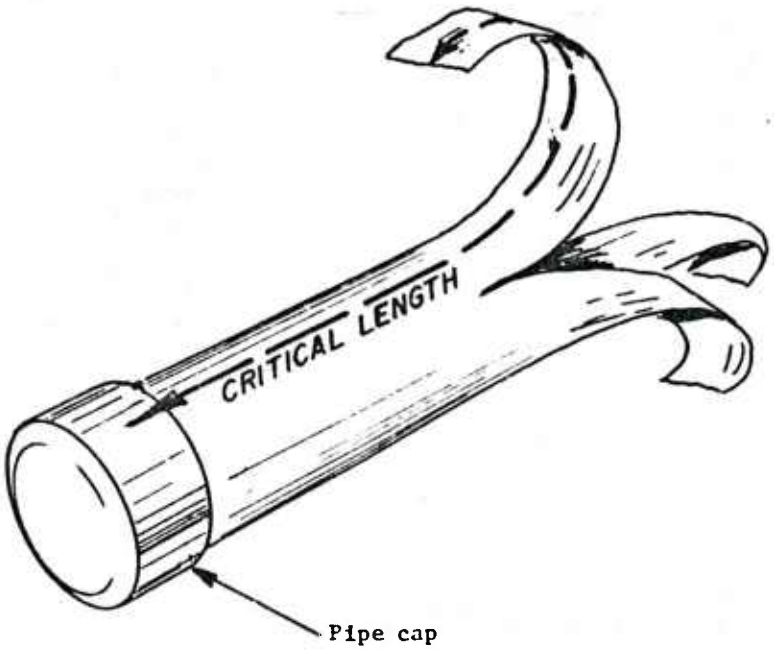


Fig 38 Typical fragments from successful critical length test

The information obtained using a continuous velocity probe to determine critical length is shown in Figure 39. The voltage signal is directly proportional to the location at which the tube is just being crushed onto the resistance wire within it. While the reaction is intensifying, the probe is "slapped around" and/or temporarily crushed giving a random oscillating signal. This signal is used to trigger the oscilloscope sweep. The point at which a detonation begins can be clearly identified from the record and the critical length computed. As mentioned earlier, the critical length as determined from the oscilloscope record was generally less than that obtained from the fragment remains.

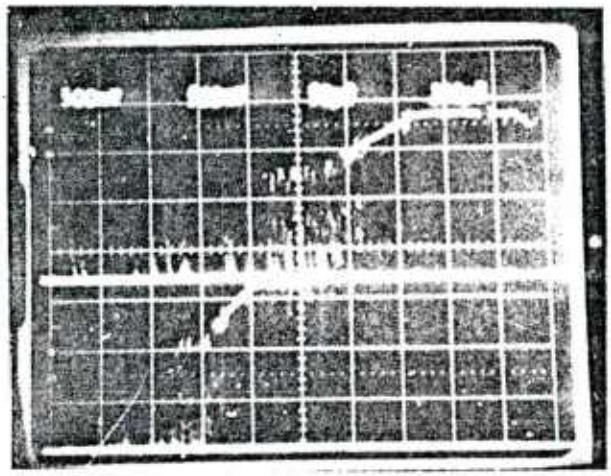
Critical Layer Thickness and Layer Transition Tests

The next two tests are completely analogous to the critical diameter and tube transition tests, except they are relevant to materials which exist in layers rather than in "bulk" configurations. The "critical layer thickness" test is analogous to the critical diameter test and the "layer transition" test is analogous to the tube transition test. In each case, much of the basic principles and philosophy discussed earlier for the "bulk" configurations still apply and will not be repeated.

Since the M30 pellets are found in a drying process in a layer, the transition tests conducted in tubing or pipe sections are not relevant for the M30 sample. To determine whether or not the pellets can propagate a detonation in the layer configuration, critical layer thickness tests were required. In these tests it was found that a detonation might be propagated in a 7.6 cm (3 in) deep layer. This was a borderline test result. Because of this result, a layer transition test was also conducted in order to determine whether a burning reaction can develop into a detonation in the first place. It was found that a 7.6 cm (3 in) deep layer of M30 pellets could not develop a detonation from a flame ignition source within the 1.37 m (4.5 ft) test length.

Critical Layer Thickness Tests

The critical layer thickness test arrangement is shown in Figures 40, 41 and 42. Four 2.54 cm (1 in.) thick steel witness plate were positioned in the field and provided a rigid bottom surface. Angle irons were used as side wall for the trough. A triangular C4 explosive booster (nearest to the camera in Figure 40) was used to develop a fairly flat detonation wave. A shorter rectangular block of C4 further flattened the detonation front before it reached the M30 pellets in the trough. To prevent the shock wave from merely throwing the pellets out at the initiating end, a short steel plate covered the pellets at the booster. Pellets filled the trough approximately flush with the top of the side walls. The first critical depth test involved pellets in a 7.6 cm (3 in.) deep layer. The witness plate remains are shown in Figure 43. The detonation clearly weakened in intensity as it travelled farther from the booster. The last witness plate was only slightly bowed. Based on witness plate damage and reaction front velocity profile, it could not



Typical oscilloscope record
(two traces with time axis doubled in bottom trace)

Note: Voltage corresponds to probe resistance since a constant current source is used. Probe resistance is directly proportional to probe length.

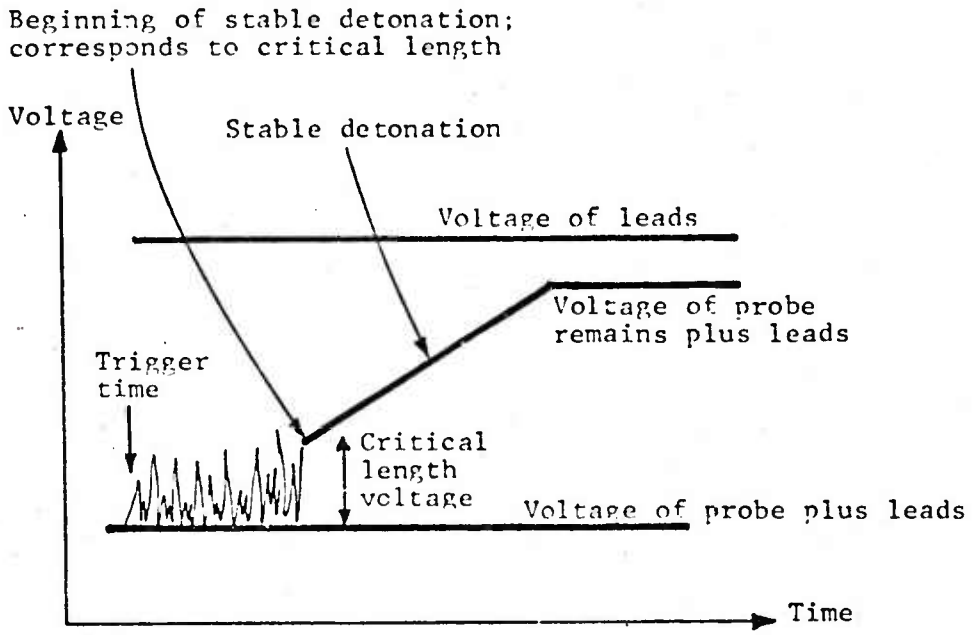


Fig 39 Ideal record from continuous velocity probe

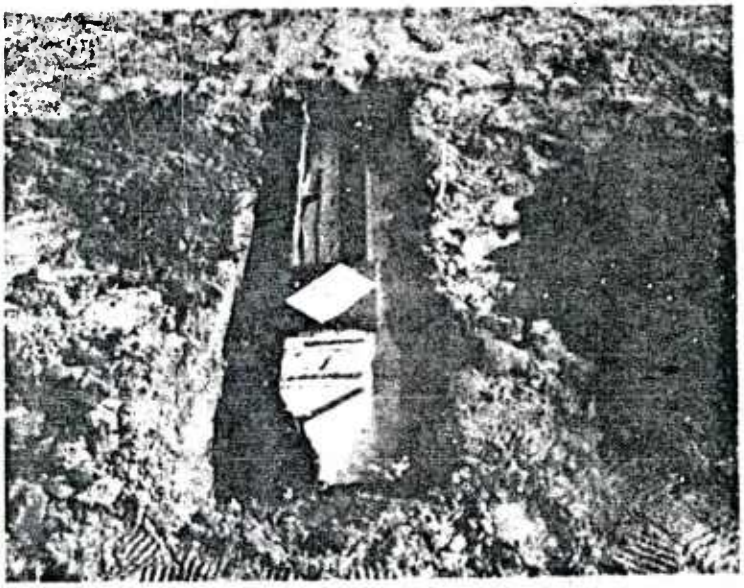


Fig 40 Critical depth test arrangement (view 1)

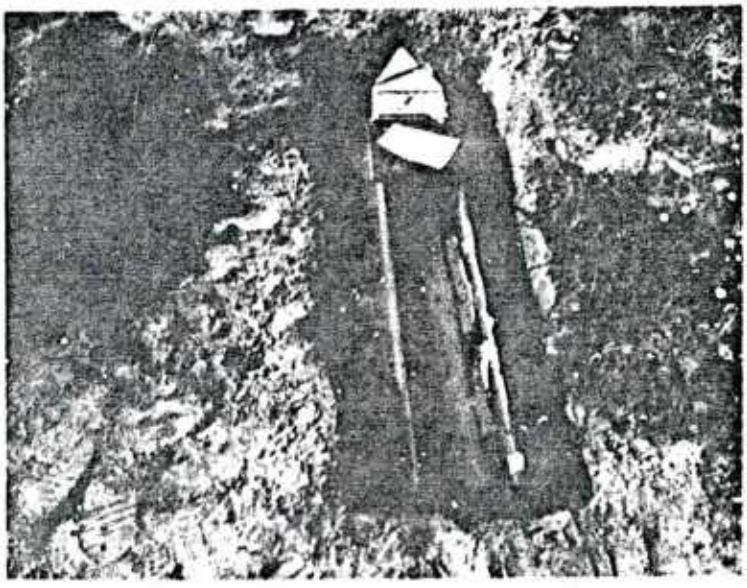


Fig 41 Critical depth test arrangement (view 2)

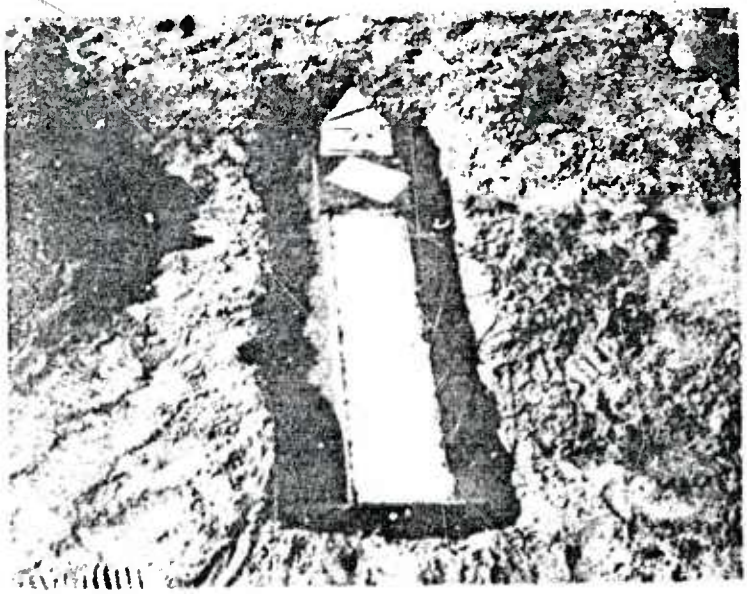


Fig 42 Critical depth test number 1 filled

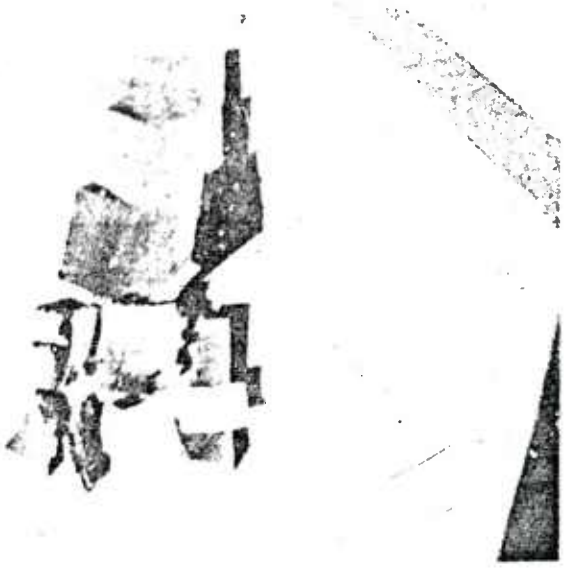


Fig 43 Critical depth test number 1, posttest witness plates

definitely be established that the detonation would have died in a longer distance. The test gave a borderline result and was therefore considered to be a "Go" to assure conservatism. The second critical depth test involved a 5.1 cm (2 in) deep layer of pellets. The witness plate remains for this test are shown in Figure 44. Unburned pellets were found scattered in the field and the last witness plate remained flat. This test was clearly a "No Go".

Layer Transition Test

Since a 7.6 cm (3 in) layer of M30 pellets might be able to propagate a detonation, a layer transition test was required to help determine whether or not a detonation could develop in the first place from a flame ignition source. The layer transition test configuration is shown in Figure 45. A steel trough 7.6 cm (3 in) deep by 15.2 cm (6 in) wide by 1.37 m (4.5 ft) long held the sample. Ignition occurred at the end nearest to the camera in Figure 45. A gas burner was used to ignite a group of pellets which in turn ignited the sample material in the trough. Reaction front velocity was observed using a continuous velocity probe at the bottom of the trough and a series of eight light sensors along the length of the trough. A detonation did not develop, therefore the continuous velocity probe did not give a signal. The light sensor data is presented in Figure 46. The reaction front velocity is seen to be increasing but a detonation is not achieved within the 1.37 m (4.5 ft) trough length. Even though the velocity is increasing, detonation is characterized by velocities on the order of 2000 m/s or greater, which is about four orders of magnitude above that measured in this test. It appears quite unlikely that a detonation could develop even given a larger trough. Thus, the most likely consequence of a small flaming ignition in a layer of M30 pellets is the spread of fire. It is also possible that a detonation originating in an adjacent equipment item could propagate through a layer of M30 pellets which is 7.6 cm (3 in) deep or more.

Mass Explosion Test

This test is to characterize the effects of the mass explosion of the sample material in its process container. The test as described here is quite similar to the "TNT equivalency" tests which have been conducted on many energetic materials for safe design and layout of process plant structures. "TNT equivalency" tests generally model the actual process vessel configuration. The container geometry is quite important to determine near field air blast effects and tests incorporating container geometry scaling will be the most realistic in terms of blast effects.

Under this program, a simpler approach was evaluated. Instead of modeling the actual process vessel geometry, a hemispherical container at ground level was used to determine a geometry independent "energy" TNT equivalency. This approach is adequate for hazards classification

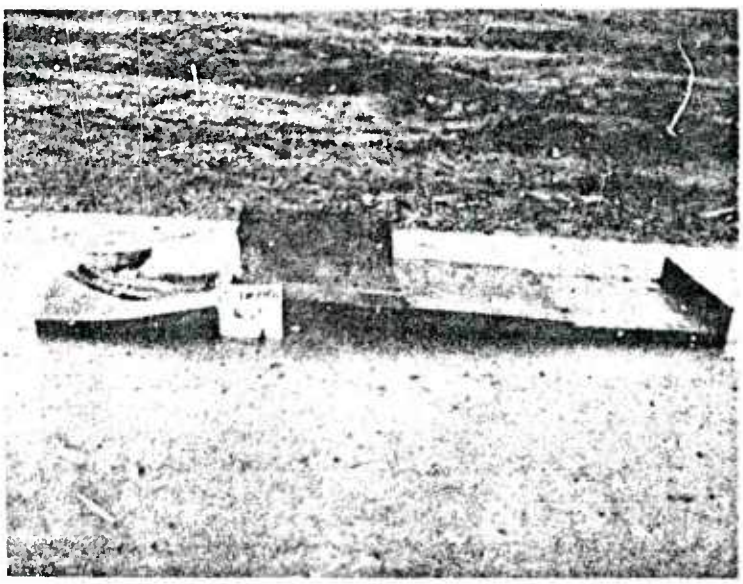


Fig 44 Critical depth test number 2, posttest witness plates

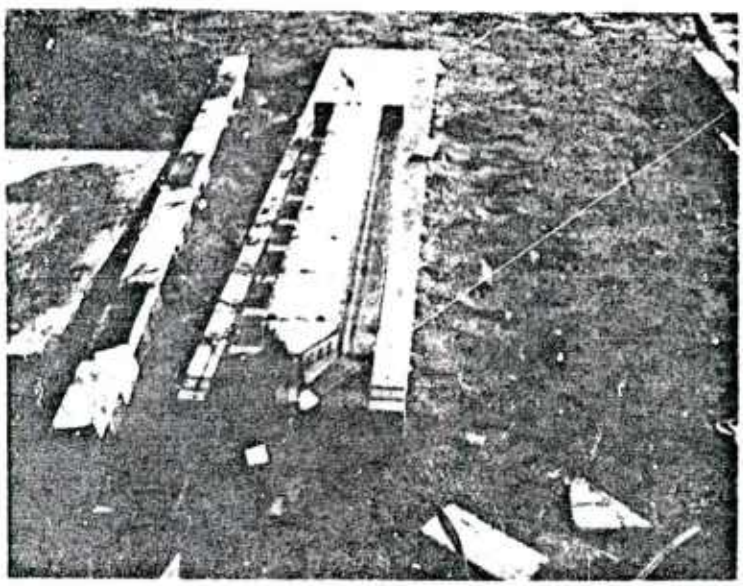


Fig 45 Critical length in layer test arrangement

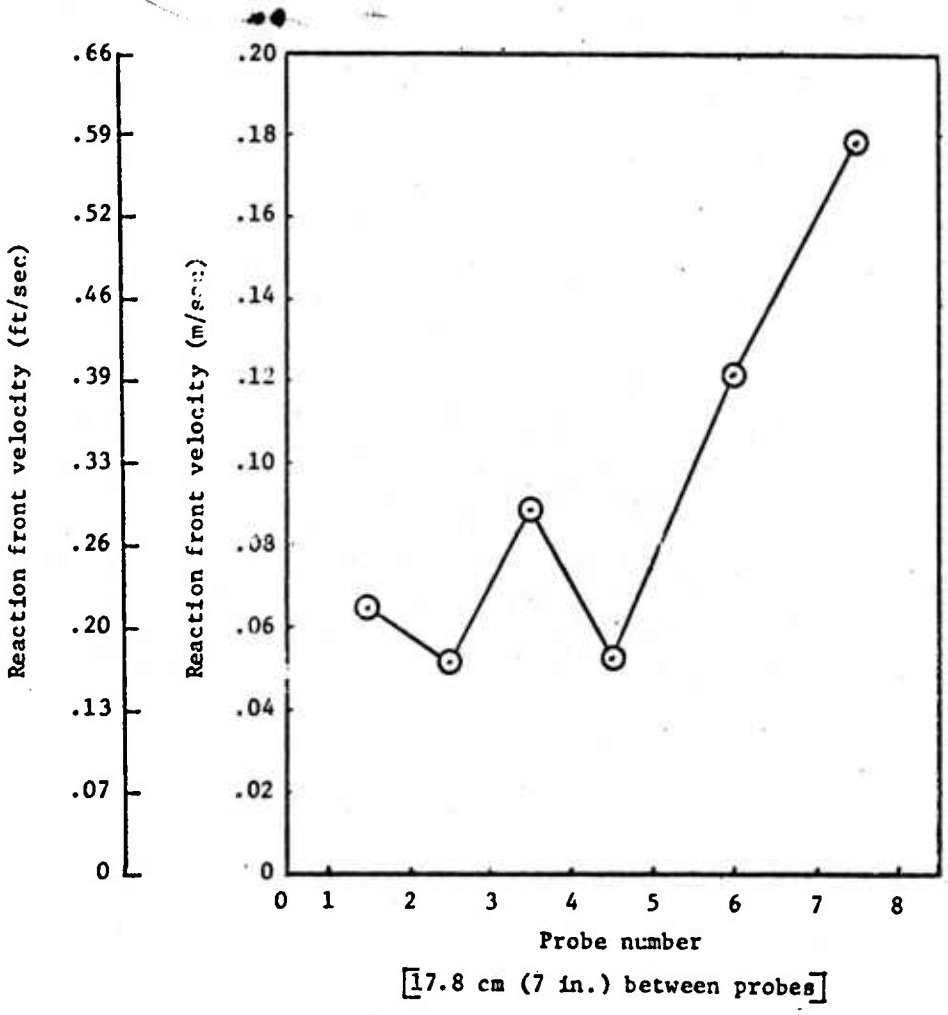


Fig 46 Reaction front velocity profile for critical length in layer test

purposes, but airblast data from tests conducted with geometric scaling are somewhat more accurate and are naturally also acceptable. For hazards classification, slug calorimeters were also positioned in the field to view the fireball to measure the energy pulse emitted by the fireball. This is necessary in order to classify the fire hazard in some cases when the air blast is not significant enough to classify the material as a mass explosion hazard.

The configuration used in these tests is illustrated in Figure 47. The test sample is packed in a thin walled hemispherical steel shell at its inprocess bulk density. Initiation is accomplished using a hemispherical C4 explosive booster as shown in the figure. Loading is accomplished by: (1) pouring a weighed out quantity of the sample into the hemisphere, (2) pushing a void for the booster into the sample, (3) positioning the booster and thin masonite sheet onto the hemisphere and (4) taping the masonite to the steel shell. The assembly is placed on a 2.54 cm (1 in.) thick steel witness plate in the field. Two perpendicular strings of pressure transducers (six pressure transducers per "leg") are arranged in the field to measure overpressure versus time for scaled distances from about 1 $m/kg^{1/3}$ (2.5 $ft/lb^{1/3}$) to about 12 $m/kg^{1/3}$ (30 $ft/lb^{1/3}$). Fastax film coverage is used to record fireball size and duration. Slug calorimeters at two radial distances from the explosion center are used to measure total heat radiated from the fireball.

The sixteen tests conducted in this program are listed in Table 20. An attempt was made to evaluate the effects of sample mass, booster percent, and steel shell wall thickness using as few tests as possible. Two tests (Numbers 5 and 14) used a weak flaming blackpowder initiation source. As anticipated, detonation did not occur in these tests. A high explosive booster is required. A successful mass explosion test involves the entire sample in the detonation process. These tests are idealized in that it is assumed that the buildup to detonation involves a negligible portion of the total material. In real explosive incidents, with large volume process vessels, this is often the case.

Three types of hazard exist in explosion incidents. Damage can be done to structures and personnel by the action of the blast wave, by heating from the fireball, and by fragments. Fragments are not considered here. Any object within the fireball will experience significant heating by radiation and convection. Therefore, the maximum fireball radius is the minimum distance for separation, regardless of the other hazards imposed. The fireball can inflict damage outside of its boundary by radiative heat transfer and this effect must also be considered.

The airblast data for the fourteen tests in which detonation occurred are presented in Appendix D. Four figures are presented for each test. In each case, the first figure shows peak overpressure versus scaled distance. The second figure presents scaled positive impulse versus scaled distance. The third and fourth figures respectively give pressure and impulse TNT equivalencies versus scaled distance.

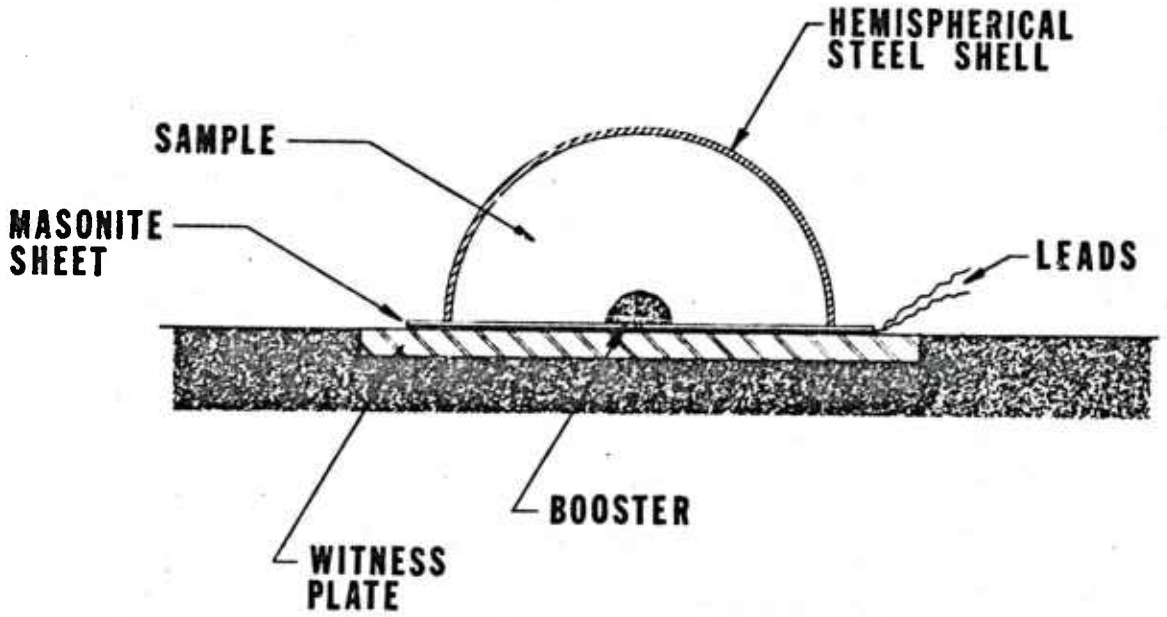


Fig 47 Mass explosion test configuration

Table 20

Mass explosion tests

Test	Sample material	Hemisphere diameter (cm)	Wall thickness (cm)	Sample mass (kg)	Booster mass (gm)	Booster percent	Booster diameter (cm)
1	M26	40	0.159	13.5	184	1.37	7.6
2	M26	40	0.159	13.3	436	3.28	10.2
3	M26	35	0.159	8.9	107	1.19	6.4
4	RDX	35	0.159	12.0	184	1.54	7.6
5	RDX	35	0.159	12.1	12 (black powder)	0.1	2.54
6	RDX	13.3	0.159	0.68	23	3.41	3.8
7	M30	40	0.159	13.6	184	1.36	7.6
8	M30	40	0.159	13.5	437	3.24	10.2
9	M30	35	0.159	7.2	107	1.18	6.4
10	M30	40	0.159	13.0	1362	10.05	14.8
11	M1	40	0.159	5.9	184	3.1	7.6
12	M1	40	0.159	5.1	436	8.5	10.2
13	M1	35	0.159	4.0	107	2.7	6.4
14	M26	35	0.159	8.9	111 (black powder)	1.25	7.6
15	M26	35	0.079	8.9	107	1.19	6.4
16	M26	35	0.318	8.9	107	1.19	6.4

To summarize the blast wave results, each sample is discussed briefly.

RDX For RDX slurry, two tests were conducted (at 0.68 kg and 12.1 kg of sample and 3.4 percent and 0.1 percent boosters). The results were essentially the same for these tests, indicating that the simple mass and booster mass percent were sufficient (i.e., the results were not dependent on either of these factors). Both pressure and impulse TNT equivalencies peaked at about 120 percent.

M30 Four tests were conducted using M30 pellets. Size scaling was evaluated using samples of about 7 kg and 13 kg both with booster percents slightly above 1 percent. These two tests gave essentially the same results with about a 20 percent equivalency for both pressure and impulse. Booster scaling was investigated with the sample at approximately 13 kg. The maximum TNT equivalencies for these tests are presented below:

Booster Percent	1.35	3.24	10.5
Pressure Equivalency, percent	22	39	64
Impulse Equivalency, percent	19	46	63

Figure 48 shows that the equivalencies tend to level off just above the 10.5 booster percent. Therefore the TNT equivalencies for both pressure and impulse will be approximately 65 percent.

M26 The results for M26 were not quite as consistent as those for the other materials. Five M26 tests were accomplished. To evaluate the effect of the steel shell wall thickness, three tests were completed using a 8.9 kg sample and a 1.2 booster percent. The equivalencies are shown below for the three wall thicknesses tested:

Wall Thickness (cm)	0.079	0.159	0.318
Pressure Equivalency, percent	100	68	80
Impulse Equivalency, percent	120	90	130

No trend was obvious from these tests. To evaluate the sample mass effect, two tests were conducted, using a 0.159 cm shell wall thickness and 1.2 to 1.4 booster percent.

Sample Mass, kg	8.9	12.9
Pressure Equivalency, percent	100	70
Impulse Equivalency, percent	120	98

The decrease in equivalency with increasing sample mass must be due to an anomaly in one of the tests. These results are not conclusive although it is likely that we were above the critical sample mass for scaling. Two tests were accomplished with a sample mass of 12.9 kg and 0.159 cm wall thickness to evaluate the booster effect.

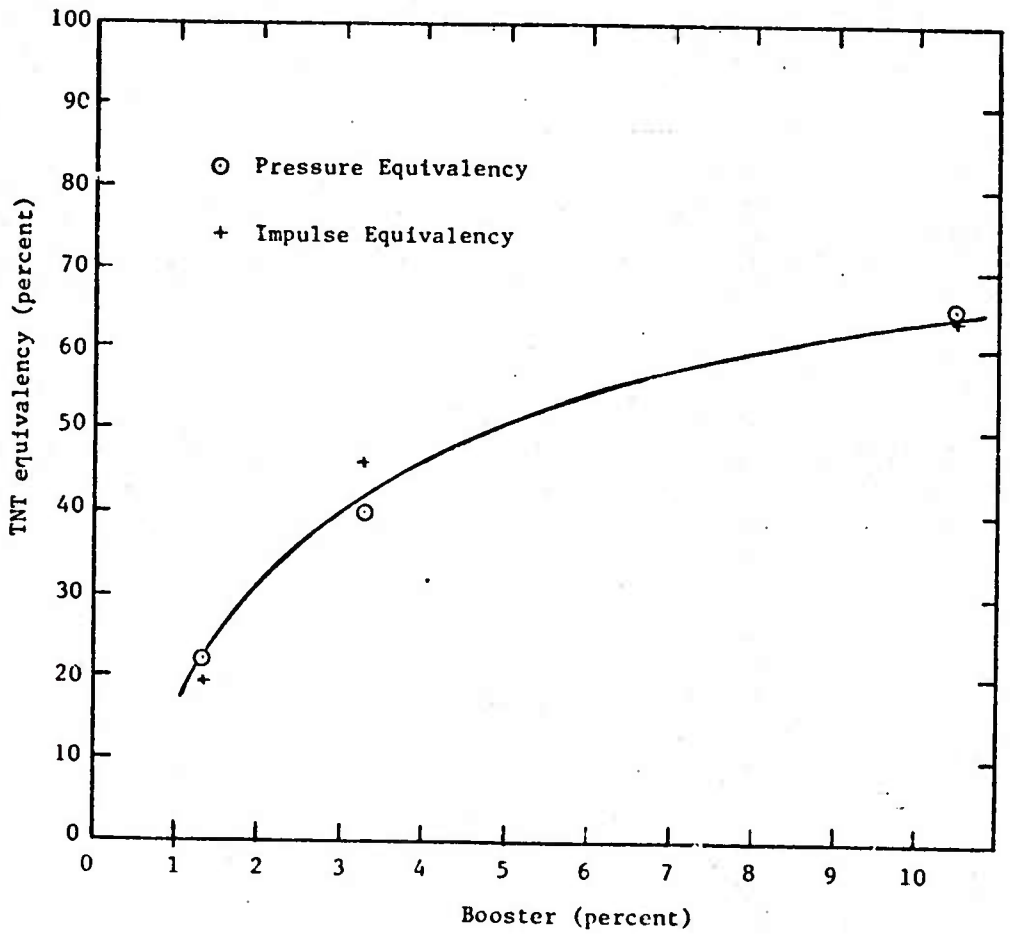


Fig 48 M30 booster scaling at 13 kg (29 lb) of M30

Booster percent	1.41	3.71
Pressure Equivalency, percent	70	80
Impulse Equivalency, percent	98	100

These results are considered to be essentially the same indicating that the results are independent of the booster percent above a value of 1.41 percent. Based on these results, it was concluded that the maximum pressure equivalency is on the order of 80 to 100 percent, and the maximum impulse equivalency is from about 100 to 130 percent.

M1 Three tests were completed with M1 strands. Each test used a different sample mass and booster percent:

Sample Mass, kg	3.98	5.13	5.92
Booster, percent	2.68	8.5	3.11
Pressure Equivalency, percent	8	22	25
Impulse Equivalency, percent	9.5	25	24

Based on these tests it is clear that the pressure and impulse equivalencies for M1 strands are both on the order of 25 percent.

As mentioned earlier, thermal effects are also evaluated in the "airblast" tests. Fastax film coverage was used to determine fireball size and duration. The fireball changes during the event and there is some question as to how to fine the fireball unambiguously. During the first millisecond (approximately), the reaction is quite concentrated, very hot and appears as a small white region on the film. This white ball grows and cools until the time at which the shock wave can be seen leaving the fireball. After the shock wave leaves, the fireball grows very slowly and appears orange on the film. The orange fireball grows to a maximum size and begins to weaken in intensity and eventually becomes obscured by smoke and debris. The fireball sizes and times at these different stages are given in Table 21 for the tests completed on this program. Insufficient testing was accomplished to show the scaling of fireball size and duration with sample mass. In general, the data was fairly random. However, based on work of this type done on previous efforts elsewhere, we expect fireball radius and duration to be proportional to the sample mass to the 1/3 power, $W^{1/3}$. From the view point of plant safety, the maximum fireball radius is the most meaningful of the choices shown in Table 21 and gave the least data spread. Assuming that fireball radius is proportional to $W^{1/3}$ we obtained the following for the four materials tested:

$R(M26) \sim 1.7\ W^{1/3}$

$R(RDX) \sim 1.5\ W^{1/3}$

$R(M30) \sim 1.36\ W^{1/3}$

$R(M1) \sim 1.47\ W^{1/3}$

Table 21

Summary of fireball characteristics taken from airblast test film coverage

Test	Sample material	Sample mass (kg)	Booster mass (gm)	White region duration (msec)	White ball radius (m)	White ball area (m^2)	Time that shock leaves fireball (msec)	Radius at which shock leaves fireball (m)	Maximum orange ball diameter (m)	Duration of orange fireball (msec)	Time to obscuration (msec)	Comments
AB-1	M26 paste	13.5	184	1.08	2.36	8.77	1.89	3.09	7.47	81.08	270	
AB-2	M26 paste	13.3	436	0.97	2.44	9.34	1.69	3.05	7.32	48.39	212	
AB-3	M26 paste	8.9	107	0.47	2.06	6.65	0.95	2.63	6.25	47.37	273	
AB-4	RDX slurry	12.0	184	0.49	2.29	8.21	0.98	2.90	5.95	24.56	130	
AB-5	RDX slurry	12.1	12 (BP)									Black powder igniter only a little white cloud of smoke observed
AB-6	RDX slurry	0.68	23	0.25	0.95	1.43	0.49	1.37	2.74	36.84	98	
AB-7	M30 pellets	13.6	184	0.49	1.03	1.67	0.95	2.21	5.95	47.71	430	
AB-8	M30 pellets	13.5	437	0.95	1.83	5.26	1.43	2.36	7.16	107.39	382	
AB-9	M30 pellets	7.2	107	0.49	0.91	1.31	0.95	1.87	5.03	47.73	134	
AB-10	M30 pellets	13.0	1362	0.95	2.10	6.91	1.67	2.90	8.54	190.91	334	
AB-11	M1 strands	5.9	184	0.25	0.73	0.83	0.74	2.06	5.34	99.82	272	Streamers
AB-12	M1 strands	5.1	436	0.47	1.11	1.92	1.65	2.06	6.16	118.64	332	
AB-13	M1 strands	4.0	107	0.50	0.88	1.21	1.00	1.34	4.88	125.00	188	
AB-14	M26 paste	8.9	111 (BP)									Black powder igniter. Nondetonation
AB-15	M26 paste (1/32" wall)	8.9	107	0.72	2.29	8.21	1.43	2.59	6.71	119.40	263	
AB-16	M26 paste (1/8" wall)	8.9	107	0.72	2.02	6.4	1.43	2.90	8.08	119.40	239	

Where W is in kilograms and R is in meters. Fireball duration was difficult to read consistently from the film record. The duration which gave the least spread of the data for any given material was the time to obscuration. Therefore, time to obscuration was selected giving:

$$\tau(M26) \sim 108\ W^{1/3}$$

$$\tau(RDX) \sim 82\ W^{1/3}$$

$$\tau(M30) \sim 156\ W^{1/3}$$

$$\tau(M1) \sim 137\ W^{1/3}$$

Where τ is in milliseconds.

To scale the thermal pulse emitted by the fireball, a simple point source model was choosen. The thermal energy per unit area, q, impinging onto a target at distance x from the source can be expressed as

$$q = \frac{M\Delta Hf}{4\pi x^2}$$

Where M is the sample mass, ΔH is the energy released in the reaction, and f is the radiated fraction of the total energy released. If we assume that for a given type of sample, the product $\Delta H \cdot f$ will be about constant, or perhaps a function of the quantity of material, the equation can be simplified to

$$q = C\frac{M}{x^2}$$

Where the coefficient C may be a function of sample mass. If tests are done using several sample masses a trend should be identifiable and extrapolation to full scale is possible.

The measured heat pulses from the mass explosion tests are presented in Table 22 with the other parameters required to compute the coefficient C. The last column gives the calculated values for C. Figure 49 shows the coefficient plotted versus sample mass for all the tests conducted. This includes all four sample materials. The data clusters somewhat for any specific sample material at a given sample mass, but there is a disappointing spread in the data which could not be attributable to any parameter variation. Booster mass does not seem to order the results and distance from the source has only a minor influence. It is interesting to note that the fireball heat pulse results for the two tests using a black powder ignition source instead of a C4 explosive booster were comparable to the other results. In terms of scaling with sample mass, RDX shows a decrease in C with mass, M26 shows an increase, and M30 just shows a wider spread of the data. In all the cases, the sample mass was not varied over a wide enough range and insufficient tests were conducted to clearly identify the scaling of C with sample mass.

Table 22

Scaling of fireball energy pulse

Test	Sample	Calorimeter distance (m)	Sample mass M (kg)	Measured heat pulse q (j/m^2)	Coefficient C (j/kg)
AB-1	M26	6.1	13.5	8.989×10^3	2.48×10^4
AB-2	M26	6.1	13.3	1.26×10^4	3.53×10^4
AB-3	M26	6.1	8.9	2.05×10^3	8.57×10^3
AB-4	RDX	6.1	12.0	1.10×10^3	3.41×10^3
		9.15	12.0	8.63×10^2	6.02×10^3
AB-5	RDX (BP igniter)	6.1	12.1	1.94 to 3.89×10^3 (avg = 2.92×10^3)	8.98×10^3
		9.15	12.1	7.59×10^2	5.25×10^3
AB-6	RDX	4.57	0.68	3.29 to 5.22×10^2 (avg = 4.26×10^2)	1.31×10^4
		6.1	0.68	3.41×10^2	1.87×10^4
AB-7	M30	6.1	13.6	1.81 to 2.47×10^3 (avg = 2.15×10^3)	5.88×10^3
		9.15	13.6	1.03×10^3	6.34×10^3
AB-8	M30	6.1	13.5	5.13×10^3	1.41×10^4
		9.15	13.5	2.87×10^3	1.78×10^4
AB-9	M30	6.1	7.2	2.16×10^3	1.12×10^4
		9.15	7.2	9.31×10^2	1.08×10^4
AB-10	M30	6.1	13.0	3.85 to 7.92×10^3 (avg = 5.89×10^3)	1.69×10^4
		9.15	13.0	2.92×10^3	1.88×10^4
AB-11	M1	6.1	5.9	2.51×10^3	1.58×10^4
		9.15	5.9	1.21×10^3	1.72×10^4
AB-12	M1	6.1	5.1	3.70×10^3	2.70×10^4
		9.15	5.1	1.46×10^3	2.40×10^4
AB-13	M1	6.1	4.0	7.26 to 9.10×10^2 (avg = 8.17×10^2)	7.60×10^3
		9.15	4.0	3.88×10^2	8.12×10^3
AB-14	M26 (BP igniter)	6.1	8.9	1.81 to 3.93×10^3 (avg = 2.87×10^3)	1.20×10^4
		9.15	8.9	9.44×10^2	8.88×10^3
AB-15	M26	4.57	8.9	2.57×10^3	6.03×10^3
		6.1	8.9	2.16×10^3	9.03×10^3
		7.62	8.9	1.63×10^3	1.06×10^4
AB-16	M26	4.57	8.9	4.20 to 5.20×10^3 (avg = 4.70×10^3)	1.10×10^4
		6.1	8.9	2.00×10^3	8.36×10^3
		7.62	8.9	1.52×10^3	9.92×10^3

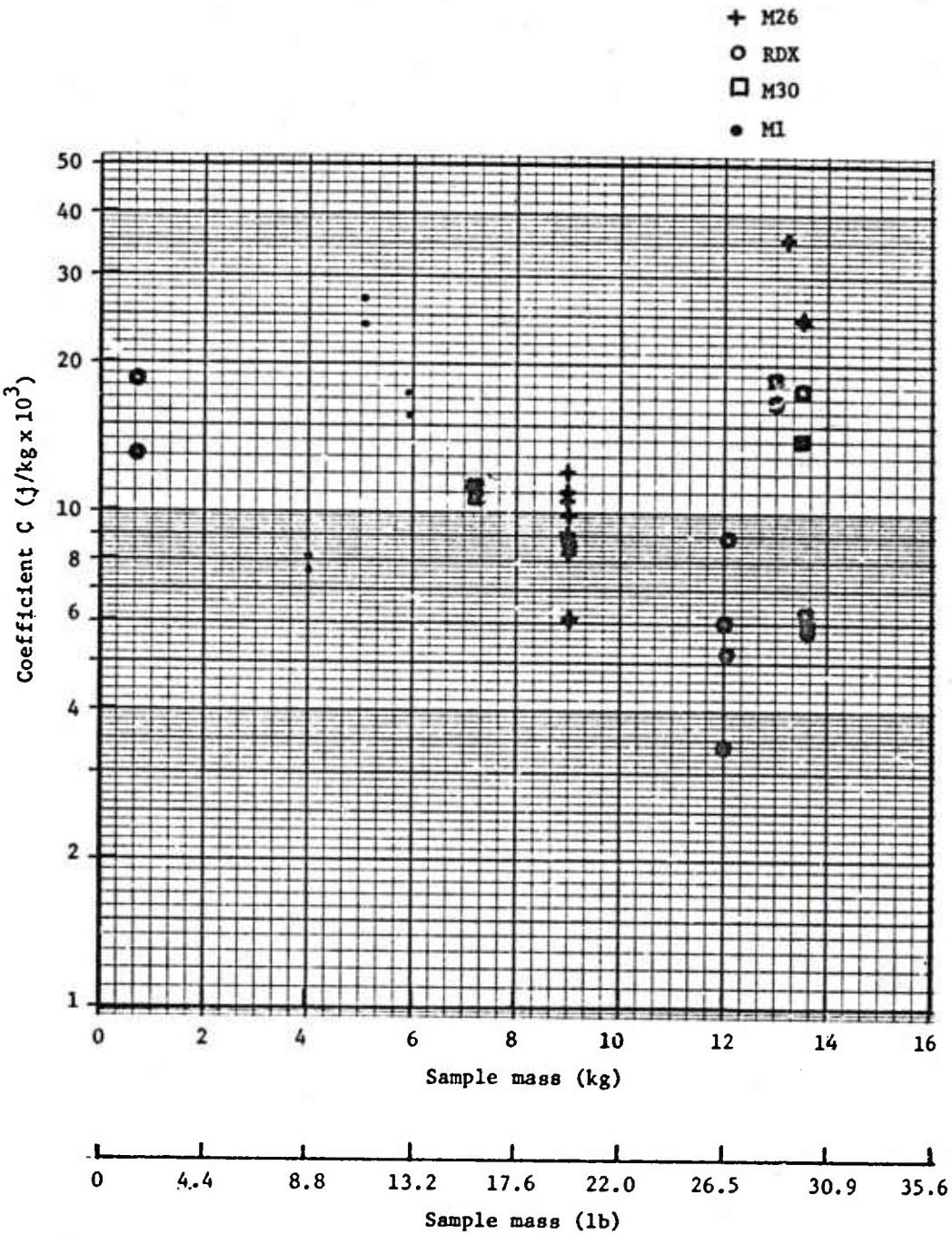


Fig 49 Coefficient for fireball energy pulse scaling

From the data we can identify values of C which may be representative of larger quantities of material. For M26 paste, the average of the two highest data points is

$$C_{M26} \sim 3 \times 10^4 \text{ j/kg}$$

If the increasing trend shown by the data would continue with increased sample mass, this value will not be high enough. For RDX sample, the average of the four data points at 12 kg is

$$C_{RDX} \sim 5.9 \times 10^3 \text{ j/kg}$$

In this case, if the decreasing trend would continue, this value would be conservative. However, based on the limited data available, we cannot be certain that the trend would not change direction with increased sample mass. The average of the four higher data points for M30 pellets give a value of

$$C_{M30} \sim 1.7 \times 10^4 \text{ j/kg}$$

The average of the four highest values obtained for M1 strands is

$$C_{M1} \sim 2.1 \times 10^4 \text{ j/kg}$$

Although the fireball test data compiled during this project gives a wide scatter in computed values of C, the point source model for scaling the radiated energy pulse is the most promising approach at the present time.

Once we are able to predict the radiated energy impinging onto a unit area of target, how do we use this information to determine whether the material represents a significant fire hazard and how do we specify safe separation distances if it is a mass fire hazard?

Ignition or damage of a target by a radiated heat pulse can be approximately accessed using a simple model that assumes ignition or damage will occur when the surface temperature of the target is raised by some critical temperature increment ΔT_c. This is not strictly correct because the heat must penetrate the surface to some depth in order to ignite or damage the target. However, the model should hold for a wide range of cases and its simplicity justifies its application.

It can be shown (Ref. 20) that the rise in surface temperature ΔT of a thick body receiving a pulse of energy per unit area, q, is given by

$$T = \frac{2q}{\sqrt{\pi\kappa\rho C'}} \frac{1}{\sqrt{\tau}}$$

Where τ is the pulse duration, κ is the materials thermal conductivity, ρ is mass density, and C' is specific heat.

This equation can be rewritten to give the critical thermal energy per unit area q_c required to raise the target's surface temperature a critical amount ΔT_c for ignition or damage within a time τ

$$q_c = \xi\sqrt{\tau} \quad \text{where} \quad \xi = \frac{\Delta T_c \sqrt{\pi\kappa\rho C'}}{2}$$

The parameter ξ characterizes the ignition or damage susceptibility of the target to a thermal pulse. This parameter can represent a wide variety of possible targets near to or within a process plant. A separate investigation could be done to identify the most meaningful parameter values to use for classifying inprocess materials and ultimately for computing safe separation distances. Such a study was not accomplished under this project. Instead, values typical for black powder were chosen to define the critical heat flux. The ignition temperature of black powder for a short duration stimulus is about 510°C (ΔT_c~490°C), the density of a black powder grain is about 1.8 gm/cm^3, and the specific heat is about 0.2 cal/gm°K (Ref. 21). The thermal conductivity of black powder was not known. It is assumed that the graphite coating will dominate the black powder thermal conductivity but the overall value is expected to be lower than graphites. Flake graphite has a thermal conductivity of 1 cal/sec cm°K at about 60°C. We assumed the black powder value will be about half this number in the calculations. Using these values, the critical energy pulse for black powder is about 7.71 x 10^6 $\sqrt{\tau}$ j/m^2 (i.e., ξ = 7.71 x 10^6 j/m^2-s$^{1/2}$). In the hazard classification procedure, the sample will be classified as a mass fire hazard if the fireball radius exceeds 3 meters or if the fireball produces this quantity of radiated thermal energy within 3 meters of the process vessel. Values of C and τ are to be derived from the mass explosion test.

To illustrate this, consider the M26 paste data generated under this project. Assume that the process vessel contains 500 kg of M26 paste. Based on relations given earlier in this section, the fireball radius would be about 13 m. Already, the material would be classified as a mass fire hazard. Suppose however that the fireball were smaller and that we have to consider the heat pulse from the fireball. The fireball duration would be estimated to be 0.86 seconds. The critical heat pulse required to ignite black powder over this duration is 7.15 x 10^6 j/m^2. The coefficient for scaling the fireball heat pulse is 3 x 10^4 j/kg. Therefore, at 3 meters from the target we would expect a target to experience about

$$q = (3 \times 10^4 \frac{j}{kg}) \frac{(500 \text{ kg})}{(3 \text{ m})^2} = 1.67 \times 10^6 \text{ j/m}^2$$

This is near to, but below, the required critical heat pulse. These results are not inconsistent in any way. They just say that the fireball size is a much more dominant influence than the heat radiated from the fireball to its surroundings in this case.

Mass Fire Test

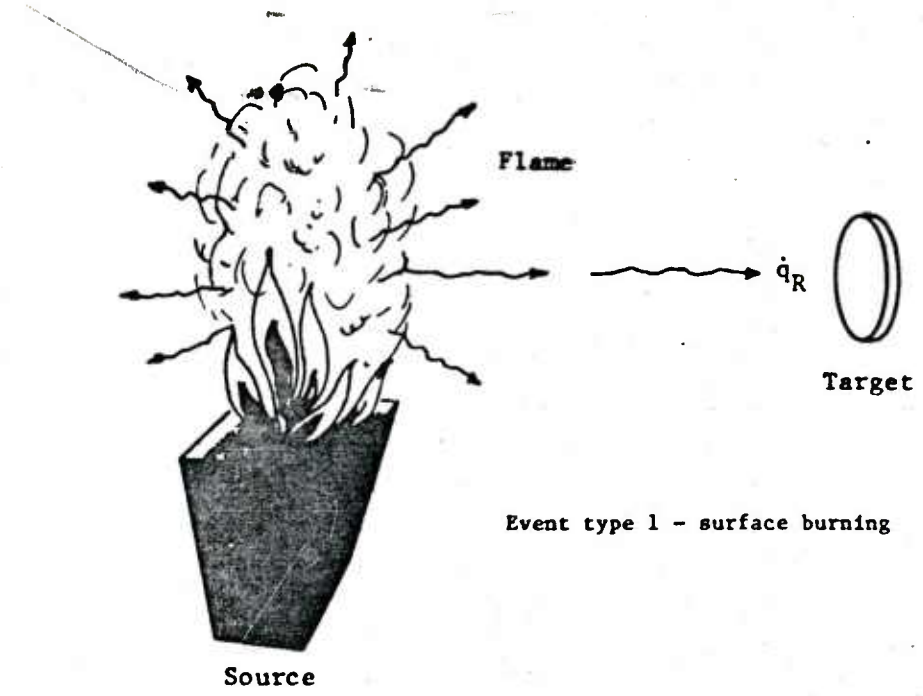
The mass fire test is done when the following conditions are met:

1. the material exists in the process operation in an open container (not a closed pressure vessel) and in a bulk configuration (not a layer or cloud)
2. the critical diameter or critical length tests indicate that the material is not likely to detonate if ignited.

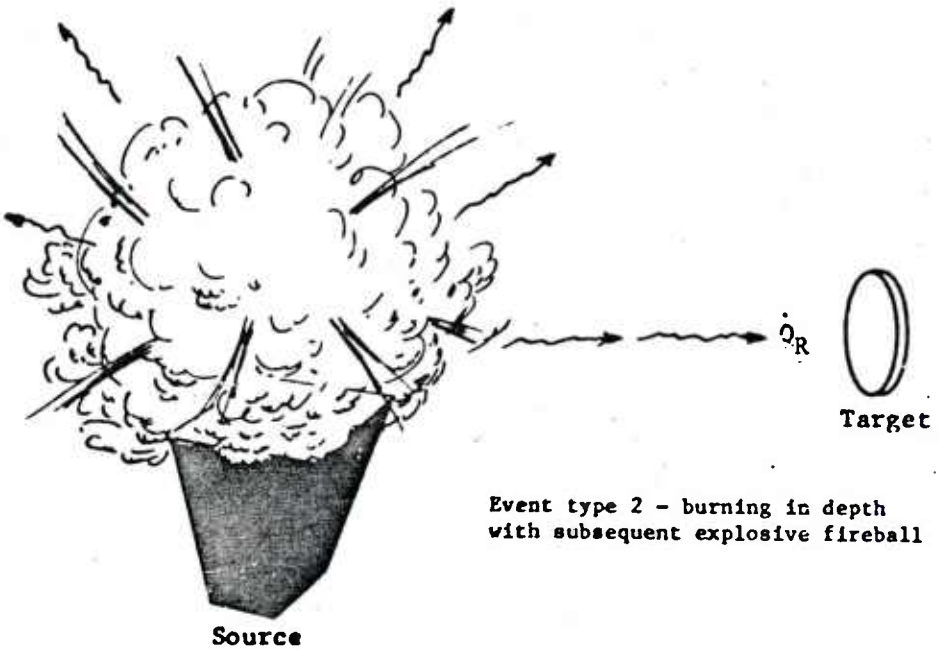
In other words, the mass fire test is done when it is likely that the consequence resulting from an ignition of the material in its process container will be a stationary fire, not an explosion or fire spread on the surface of a layer of material. Since many process materials carry their own oxidizer, burning within the bulk of material accompanied by a pressure buildup and resultant fireball is also possible. The purpose of the test is to determine the separation distances required between the vessel being evaluated and other process vessels, other process buildings and occupied buildings in order to prevent firespread or personnel injury by remote radiative heat flux.

Pertinent Theoretical Background

The physical problem being evaluated by the mass fire test is shown in Figure 50. Material in an open topped container is ignited and burns in the container in one of two ways. If the burning process proceeds relatively slowly a fire plume will emerge from the top surface; the burning will proceed analogous to a candle burning (Type 1 bulk burning event). This situation is expected to be by far the most common for materials which are not routed to the mass explosion test. Alternately, the burning can proceed quickly in depth below the surface. In this case, it is possible that the reaction below the surface will produce pressures sufficient to blow out the reacting material generating a fireball (Type 2 bulk burning event). Without more experience in conducting this type of test, we cannot predict with certainty before a test which phenomena will occur. Therefore, we must be prepared to collect the pertinent data for both types of events in a bulk burning test. To define the pertinent data for the two types of events, each event is discussed further below.



Event type 1 - surface burning



Event type 2 - burning in depth
with subsequent explosive fireball

Fig 50 Mass fire physical problem

The first type of event is the stable steady burning of the process material down from its top surface. This burning behavior will produce a fire column above the top surface of the material in its container. The fire column will radiate thermal energy in all directions. To identify the dominating parameters, the radiative heat flux from the fire column can be described in the following way.

We know that a quantity of energy per unit mass, ΔH_c, is stored chemically in the sample. Depending on how efficiently the combustion process proceeds, a fraction f_1 of this energy is released in the chemical reaction. Some of this released energy is convected upward with the hot gases in the fire column and some (a fraction f_2) is radiated in all directions from the fire column. The fraction of energy radiated depends on the flame properties (its absorption coefficient and thickness). Of the radiated energy, we are interested in the fraction which impinges on the target. For a distant* target the fraction of the radiant energy impinging on the target will be $A/4\pi R^2$ where A is the target surface area and R is the distance from the source to the target. Thus, for this type of event, the radiant heat flux per unit target surface area, q_r, will be

$$q_r = \frac{1}{4\pi R^2} f_1 f_2 \Delta H_c \dot{m}_v$$

Where $\dot{m}_v$ is the rate of mass consumption during the combustion process, which is the same as the sample weight loss rate. Thus, in experimentally evaluating a "Type 1" bulk burning event the following points should be considered:

- The material depth in the container can influence burning rate per unit surface area. Therefore, several depths should be tried to assure that the depth is sufficient so that the material acts as though it were infinitely deep or at least as deep as in the actual process vessel. Burning rate (weight loss rate) must be measured to evaluate this effect.
- The breadth of the sample can influence burning rate per unit area by cooling at the outer diameter. Therefore, several breadths should be tried to assure that the boundary influence is no longer significant. Weight loss rate should be measured here also.
- Flame thickness and the effective absorption coefficient will control the fraction of energy radiated from the flame. We will assume that burning any material will produce fairly consistent fire products (smoke gases and

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* Distant implies that the flame is small compared to the distance, i.e., the flame looks like a point source of radiant energy.

particulates). Therefore, flame emissivity will be controlled primarily by the flame thickness. Several fire diameters should be tried to evaluate this influence. However, there is no need to have a diameter greater than exists in the actual process vessel.

The second type of bulk burning event results in a fireball. The concepts for this case are essentially the same as those discussed already for the mass explosion test fireball and will not be repeated here.

Experimental Evaluation

The best candidate sample material for evaluating this test was the M1 strands. Four mass fire tests were conducted using M1 strands in metal cylindrical containers (length to diameter ratio of 1.0) with diameters 15.24 cm (6 in), 23 cm (9 in), 30 cm (12 in) and 41 cm (16 in). Initiation was accomplished using an S65 squib in a 5 gram bag of black powder centered on the top surface of the sample material. As the sample burned; its mass was measured using a force transducer through a lever arm as shown in Figure 51.

In this type of test, we do not know beforehand how quickly the sample will burn. It can burn very quickly, building up pressure in depth within the material, throwing out the material and producing a fireball. Conversely, the material can burn relatively slowly producing a "bunsen burner" type flame sitting on the top surface.

If the reaction is quick resulting in a fireball, calorimeters must be used to measure the heat pulse's total heat emitted per unit area. In this case the event duration is obtained from movie coverage and the mass of material assumed to contribute energy is the total weight of sample present. If the reaction is relatively slow resulting in a long duration flame, radiometers must be used to measure the heat flux. For this case, the force transudcer provides the weight loss profile and flame geometry is obtained from the movie coverage.

Before conducting the tests on M1 strands, we did not know which burning behavior to expect. Therefore, we were prepared to collect data for both types of event. Both radiometers and calorimeters were present and both regular (32 frames per second) and fastax movie coverage were used. In all four tests conducted using the M1 strands, the slower "bunsen burner" type burning occurred. We expect that this will be the more common type of result for inprocess materials which are categorized as fire, rather than explosion, hazards based on the screening tests. Although if a quick "fireball" type of reaction occurs, the remote heat flux data can be analyzed in a manner identical to that which was done in the mass explosion tests.

Since our test results all involved the longer duration burning, the discussion which follows is oriented toward that type of event. The purpose of the investigation which was conducted was to identify the most

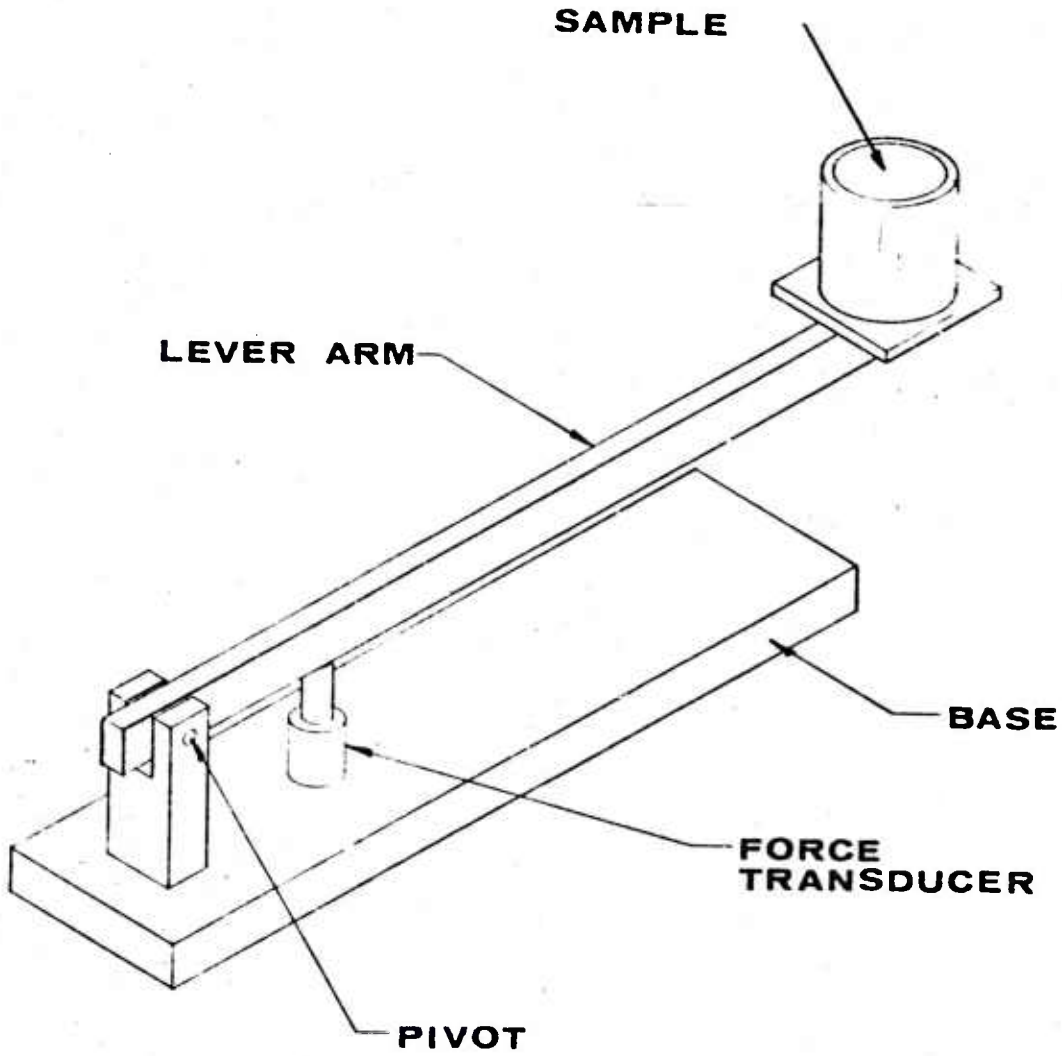

Fig 51 Mass fire test arrangement

promising technique of correlating the data so that remote heat flux produced in full scale process fires can be extrapolated (scaled) from small scale test data. Two approaches of correlating the data were investigated. The first approach (Approach 1) is based on the remote heat flux being proportional to the distance from the flame squared, L^2:

$$\dot{q} = C\frac{\dot{m}}{L^2} \quad \text{where C is a constant}$$

We hoped to find that C is truly a constant or shows a clear trend as the test container size is increased. Unfortunately, in the four tests which were conducted, C was not as well behaved as was hoped. This was probably due to an anomaly in the fourth test and does not necessarily invalidate the technique.

The second approach which was tried (Approach 2) considers the flame as an area source of heat; it was assumed that a well behaved or constant effective flame temperature, T_f, can be defined for any sample material and that variations in remote heat flux can be explained based on a simple radiative heat transfer model of the form:

$$\dot{q} = \underbrace{\left(1 - e^{-\alpha d}\right)}_{\varepsilon_f} F\sigma \left(T_f^{\,4} - T_a^{\,4} \right)$$

Where ε_f is the flame emissivity, α is the absorption coefficient, d is the flame diameter, F is the configuration factor as given in Figure 52 (a function of flame shape and distance to the target), σ is the Stefan-Boltzmann constant and T_a is the ambient temperature. Using approach 2 the results seem to scale somewhat better for the available data.

To evaluate approach 1, the constant C was computed from the experimental data at selected times after initiation. The results of these calculations are given in Table 23. The value of C was found to be fairly constant at any time for each test (i.e., independent of target distance) showing that distance squared scaling is reasonable. In most cases, C was even fairly constant over time for any individual test. The constant did change however for the different tests showing a dependence on container diameter. This dependence is plotted in Figure 53. The shape of the curve and particularly the extreme drop in C at the 41 cm (16 in.) diameter was unexpected and should be verified with more tests if scaling by approach 1 is to be used. The 41 cm diameter point is questionable since a quantity of sample material was thrown out of the container about 15 seconds after the test began. After 15 seconds a pile of burning material lay next to the test container. Two large flames of about equal intensity continued to burn next to each other for the remainder of the test.

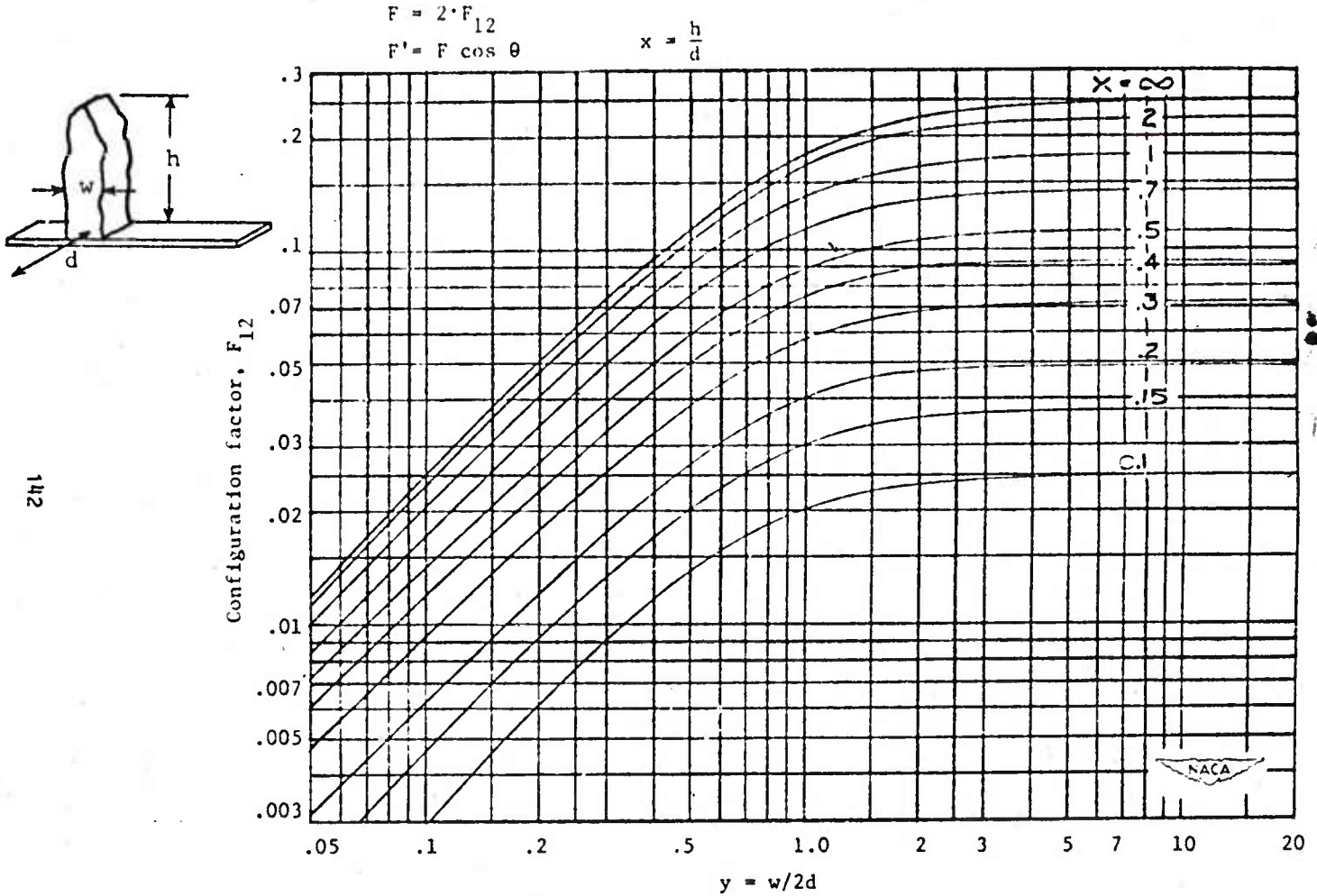

Fig 52 Configuration-factor curves (taken from NACA TN2836)

Table 23

Estimation of the constant C for approach 1

Test	Time (sec)	Distance (m)	Heat flux (w/cm^2)	Weight loss rate (gm/sec)	C $\left(\frac{1}{gm}\frac{m^2}{cm^2}\right)$	Average C $\left(\frac{1}{gm}\frac{m^2}{cm^2}\right)$
BIB-1	5	1.52	0.260	∿ 22.16	0.027	
diameter = 15.2 cm	5	3.05	0.082	∿ 22.16	0.034	
W = 1.25 kg	31	1.52	0.539	∿ 22.16	0.056	0.067
	31	3.05	0.165	∿ 22.16	0.069	
	38	1.52	0.640	∿ 22.16	0.067	
	38	3.05	0.178	∿ 22 16	0.075	
BIB-2	7	4.51	0.069	12.98	0.112	
diameter = 922.9 cm	7	6.10	0.039	12.98	0.111	
W = 4.21 kg	12	4.57	0.067	12.98	0.108	0.095
	12	6.10	0.036	12.98	0.104	
	24	4.57	0.201	62.65	0.067	
	24	6.10	0.117	62.65	0.069	
BIB-3	10	6.10	0.068	0		
diameter = 30.4 cm	10	9.15	0.024	0		
W = 9.99 kg	22	6.10	0.156	59.02	0.092	
	22	9.15	0.056	59.02	0.074	
	31	6.10	0.138	59.02	0.082	0.083
	31	9.15	0.051	59.02	0.091	
	44	6.10	0.227	102.15	0.083	
	44	9.15	0.096	102.15	0.079	
BIB-4	5	6.10	0.149	322.34	0.017	
diameter = 40.64 cm	5	7.62	0.073	322.34	0.013	
W = 23.61 kg	5	9.15	0.065	322.34	0.017	
	5	12.20	0.034	322.34	0.016	0.018
	10	6.10	0.175	322.34	0.020	
	10	7.62	0.109	322.34	0.020	
	10	9.15	0.079	322.34	0.021	
	10	12.20	0.050	322.34[a]	0.029	
	19	6.10	0.579	(1317 + other	0.016 → 0.008	
	19	7.62	0.404	pile, ∿ 2724	0.018 → 0.009	
	19	9.15	0.306	total)	0.020 → 0.010	
	19	12.20	0 200		0.023 → 0.011	

[a] 9.53 kg of material was thrown out at 15 sec. A pile of sample burned on the gound next to the test container for the remainder of the test.

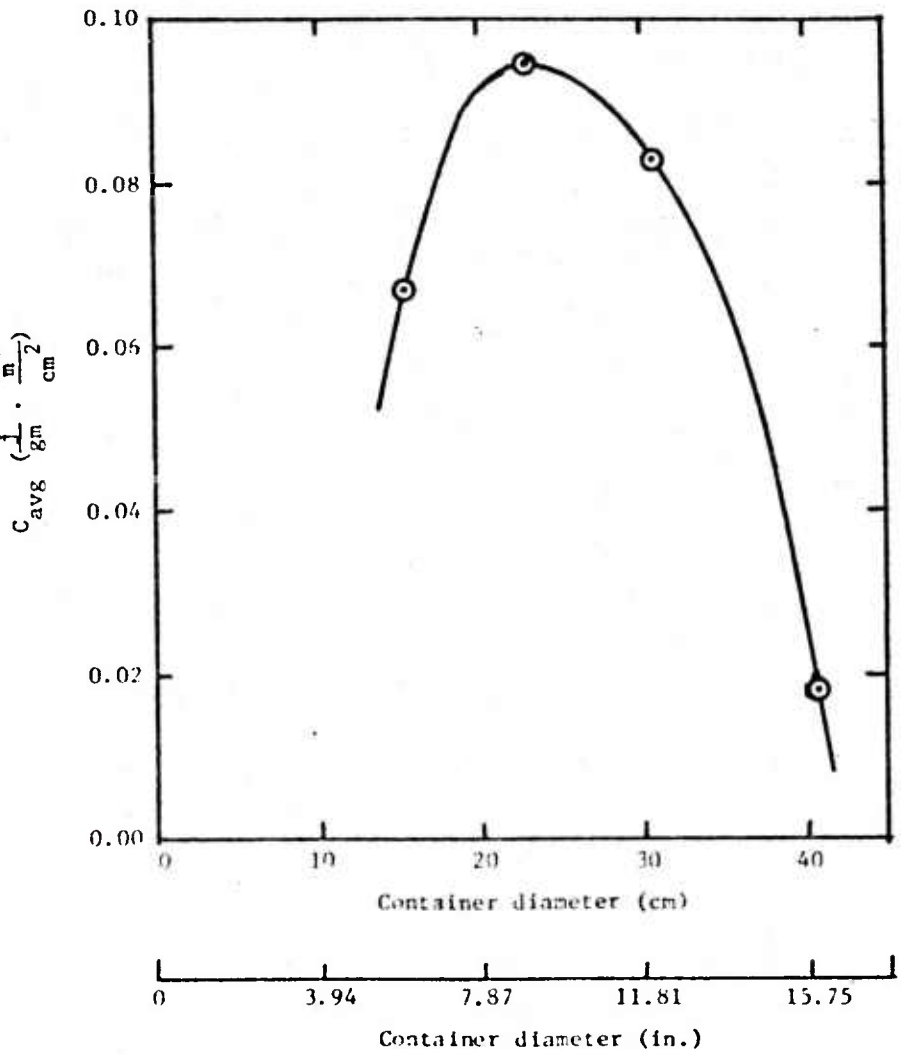

Fig 53 Constant C versus container diameter

Burning rate is also needed in order to scale q using approach 1. Burning rate (weight loss rate) is plotted as a function of container diameter in Figure 54. A fairly well behaved curve was identified and scaling of this parameter is reasonable. The major problem with approach #1 is that the parameter C is not well behaved according to our data; therefore, it would be difficult to extrapolate C to larger container sizes.

In approach 2 the effective flame temperature is computed from experimental data using the relation

$$T_f = \left(\frac{\dot{q}}{(1-e^{-\alpha d})F\sigma} + T_a^4 \right)^{1/4}$$

The calculations are summarized in Table 24 and the resultant effective flame temperature profiles are plotted in Figure 55. The maximum flame temperature from these tests is plotted versus the container cross-sectional area (sample exposed surface area) in Figure 56. The four tests are not adequate to show that all materials will scale in the same manner, but for M1 strands a clear trend is identified with the effective flame temperature decreasing and leveling off at about 815°C (1500°F) to 870°C (1600°F) at large container diameters. Thus, in the case of M1 strands, the maximum effective flame temperature seems to scale quite well.

In order to predict maximum heat flux for full scale process vessels using approach 2, we must also be able to scale ε_f and F. Both of these parameters are functions of the flame diameter and height.

The maximum flame height is plotted in Figure 57 as a function of container diameter and the ratio of flame diameter to container is plotted in Figure 58. Both flame shape parameters seem to scale reasonably well as the container size is increased. For conventional fuels, Thomas (Ref 24) showed that scaling the flame's length to diameter ratio (L/D) can be accomplished using a dimensionless burning rate parameter in the following way

$$\frac{L}{D} = \alpha \left(\frac{\dot{m}''}{\rho_a \sqrt{gD}} \right)^{\beta}$$

Where $\dot{m}''$ is the fuel generation rate per unit area, ρ_a is the density of ambient air, g is the gravitational acceleration, D is the flame base diameter, and α and β are emperically derived constants. For conventional fuels α=42 and β=0.61. For the four tests using M1 strands, a very good fit to the experimental data can be obtained by using the flame diameter instead of the container diameter with α=23.32 and β=0.65. Using the technique, the calculated observed L/D ratios compare as indicated below

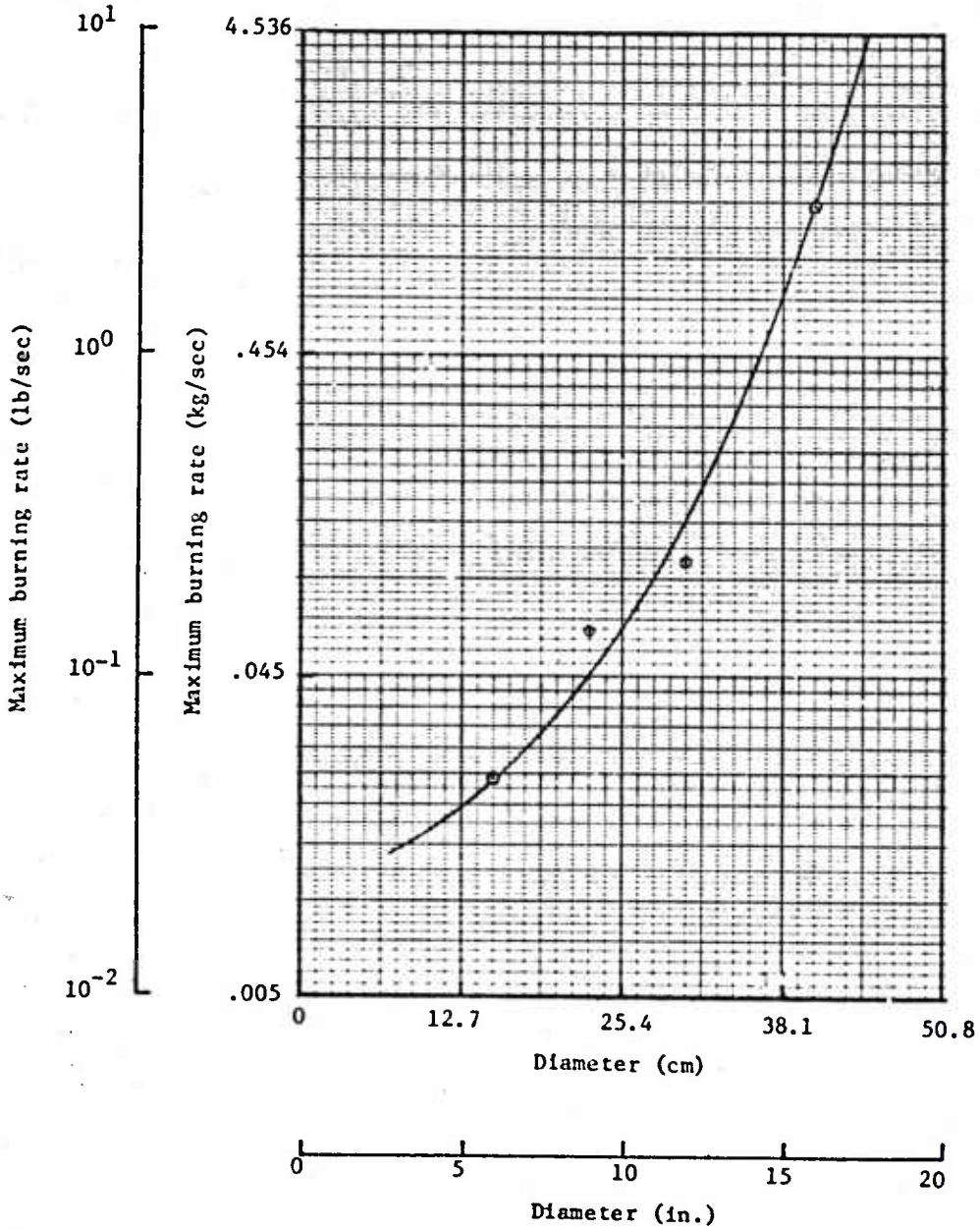

Fig 54 Maximum burning rate versus container diameter for burning in bulk tests with M1 strands

Table 24

Estimation of effective flame temperature, T_f

Test	Time	Measured heat flux		Distance		Measured heat flux at 3.05 m (10 ft)		Flame height		Flame diameter		X^a	Y^a	F	C_f	T_f	
	(sec)	W/m^2	(Btu/ft^2-sec)	m	(ft)	W/m^2	(Btu/ft^2-sec)	M	(ft)	m	(ft)					°C	(°F)
B1B-1	5	2565	(0.226)	1.52	(5)	647	(0.057)	1.07	(3.50)	0.41	(1.33)	0.700	0.266	0.043	0.285	1113	(2036)
	5	817	(0.072)	3.05	(10)	817	(0.072)					0.350	0.133	0.014		1106	(2022)
	31	5391	(0.475)	1.52	(5)	1351	(0.119)	0.91	(3.00)	0.49	(1.60)	0.600	0.320	0.047	0.332	1298	(2369)
	31	1646	(0.145)	3.05	(10)	1646	(0.145)					0.300	0.160	0.014		1308	(2386)
	38	6401	(0.564	1.52	(5)	1600	(0.141)	0.96	(3.15)	0.46	(1.50)	0.630	0.300	0.045	0.315	1407	(2564)
	38	1782	(0.157)	3.05	(10)	1782	(0.157)					0.315	0.150	0.014		1361	(2481)
B1B-2	7	695	(0.0612)	4.57	(15)	1566	(0.136)	1.83	(6.00)	0.91	(3.00)	0.400	0.200	0.0225	0.530	735	(1355)
	7	386	(0.034)	6.10	(20)	1543	(0.136)					0.300	0.150	0.014		707	(1305)
	12	670	(0.059)	4.57	(15)	1509	(0.133)	2.62	(8.60)	0.55	(1.80)	0.573	0.120	0.0135	0.365	972	(1781)
	12	363	(0.032)	6.10	(20)	1453	(0.128					0.430	0.090	0.012		827	(1521)
	24	2009	(0.177)	4.57	(15)	4517	(0.398)	2.68	(8.80)	0.91	(3.00)	0.587	0.200	0.012	0.530	1263	(2306)
	24	1169	(0.103)	6.10	(20)	4676	(0.412)					0.440	0.150	0.018		940	(1724)
B1B-3	10	681	(0.060)	6.10	(20)	2724	(0.240)	1.22	(4.00)	0.76	(2.50)	0.200	0.125	0.0075	0.467	1088	(1990)
	10	238	(0.021)	9.14	(30)	2145	(0.189)					0.133	0.083	0.0032		1022	(1872)
	22	1555	(0.137)	6.10	(20)	6219	(0.548)	1.58	(6.50)	1.07	(3.50)	0.325	0.175	0.017	0.586	1016	(1860)
	22	545	(0.048)	9.14	(30)	4903	(0.432)					0.217	0.117	0.0073		958	(1756)
	31	1385	(0.122)	6.10	(20)	5538	(0.488)	2.01	(6.60)	1.01	(3.30)	0.330	0.165	0.016	0.565	1009	(1849)
	31	511	(0.045)	9.14	(30)	4596	(0.405)					0.220	0.110	0.007		956	(1753)
	44	2270	(0.200)	6.10	(20)	4540	(0.400)	2.59	(8.50)	1.22	(4.00)	0.425	0.200	0.0235	0.636	1045	(1913)
	44	965	(0.085)	9.14	30)	8682	(0.765)					0.283	0.133	0.012		950	(1742)
B1B-4	5	1487	(0.131)	6.10	(20)	5947	(0.524)	3.51	(11.50)	0.91	(3.00)	0.575	0.150	0.022	0.530	952	(1746)
	5	726	(0.064)	7.62	(25)	4540	(0.400)					0.460	0.120	0.015		854	(1570)
	5	647	(0.057)	9.14	(30)	5822	(0.513)					0.383	0.100	0.012		885	(1625)
	5	340	(0.030)	12.19	(40)	5447	(0.480)					0.288	0.175	0.0066		872	(1602)
	10	1748	(0.154)	6.10	(20)	6991	(0.616)	3.57	(11.70)	1.16	(3.80)	0.585	0.190	0.029	0.616	874	(1605)
	10	1090	(0.096)	7.62	(25)	6809	(0.600)					0.468	0.152	0.0195		852	(1566)
	10	794	(0.070)	9.14	(30)	7150	(0.630)					0.390	0.127	0.014		857	(1574)
	10	499	(0.044)	2.19	(40)	7920	(0.704)					0.293	0.095	0.0085		867	(1592)
	19	5788	(0.510)	6.10	(20)	23,152	(2.040)	10.67	(35.00)	1.52	(5.00)	1.750	0.250	0.0560	0.716	991	(1815)
	19	4040	(0.356)	7.62	(25)	25,308	(2.230)					1.400	0.200	0.044		954	(1749)
	19	3064	(0.270)	9.14	(30)	27,578	(2.430)					1.170	0.167	0.035		939	(1723)
	19	1997	(0.176)	12.19	(40)	32,004	(2.820)					0.875	0.125	0.025		912	(1674)

[a] Parameters for estimating radiative interchange configuration factor, F, defined in Figure 50.

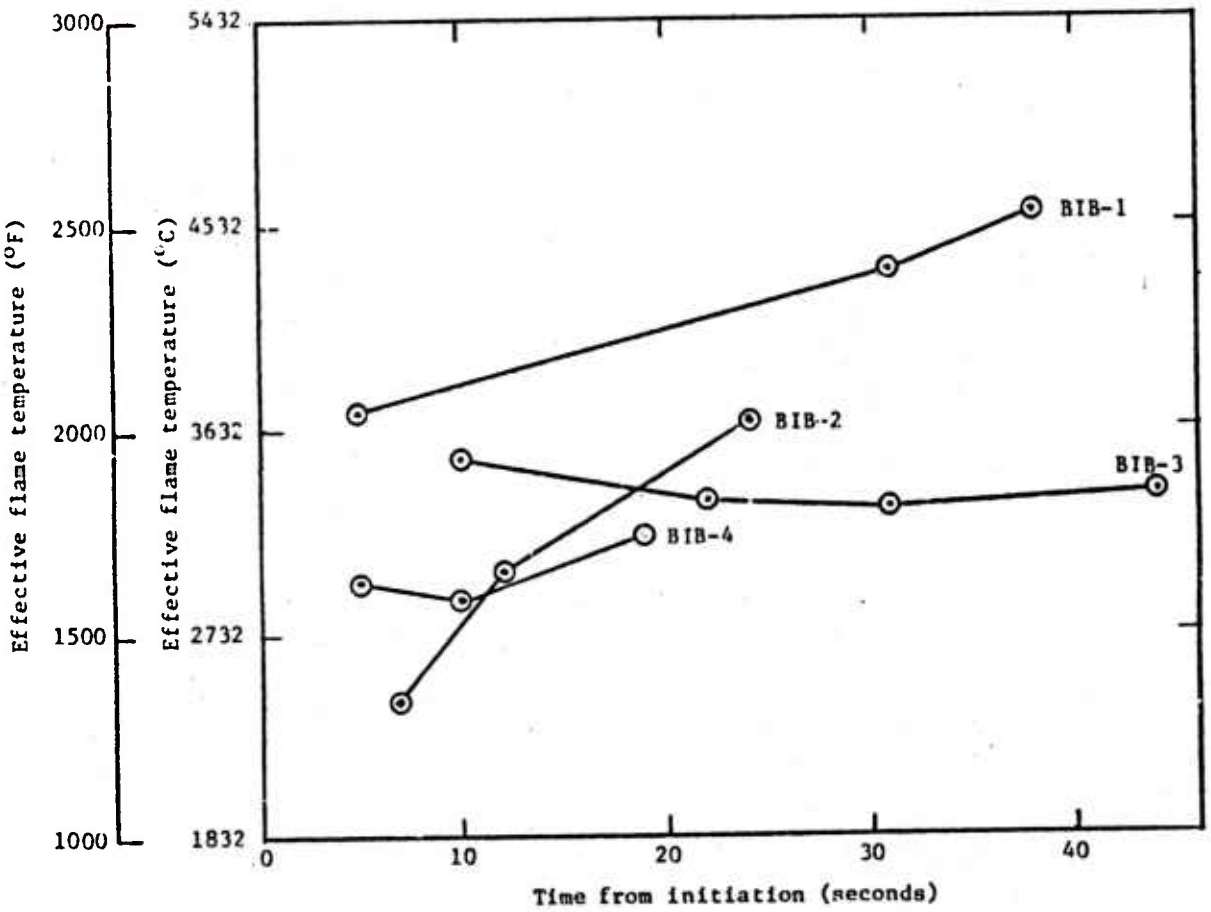

Fig 55 Effective flame temperature versus time for the burning in bulk tests

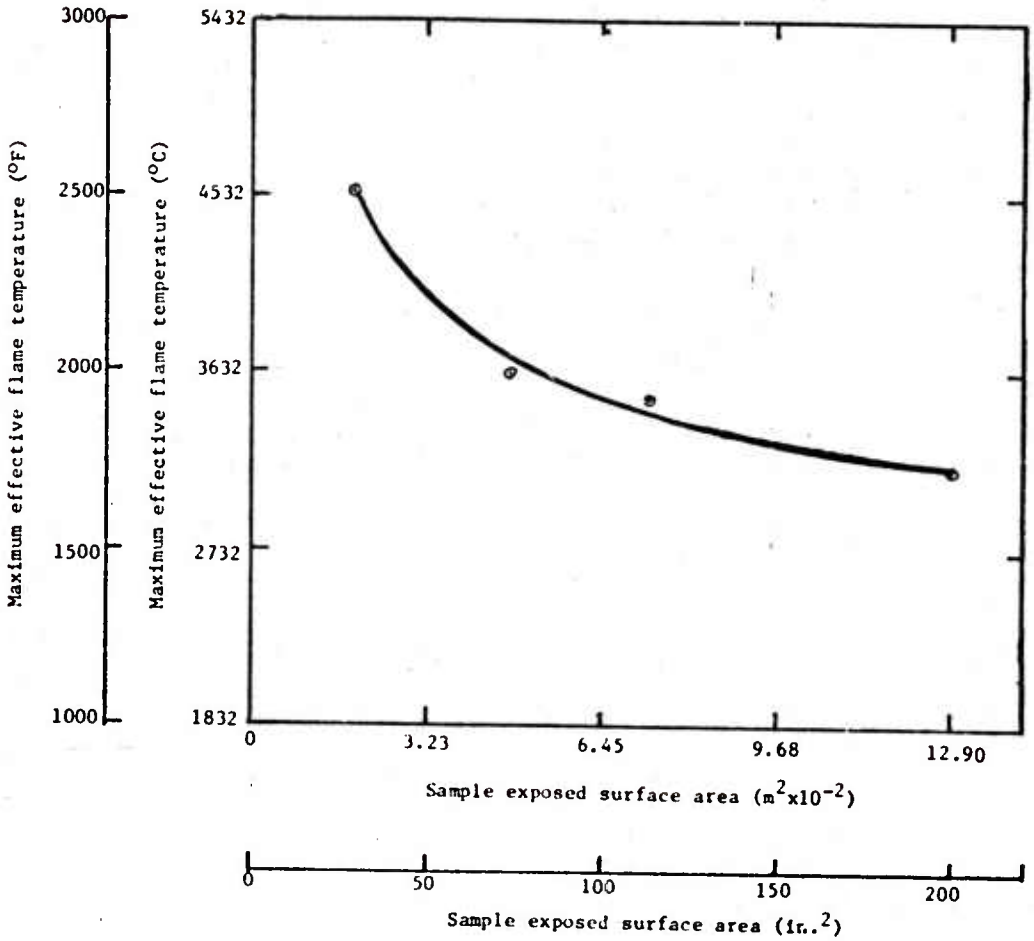

Fig 56 Scaling maximum effective flame temperature

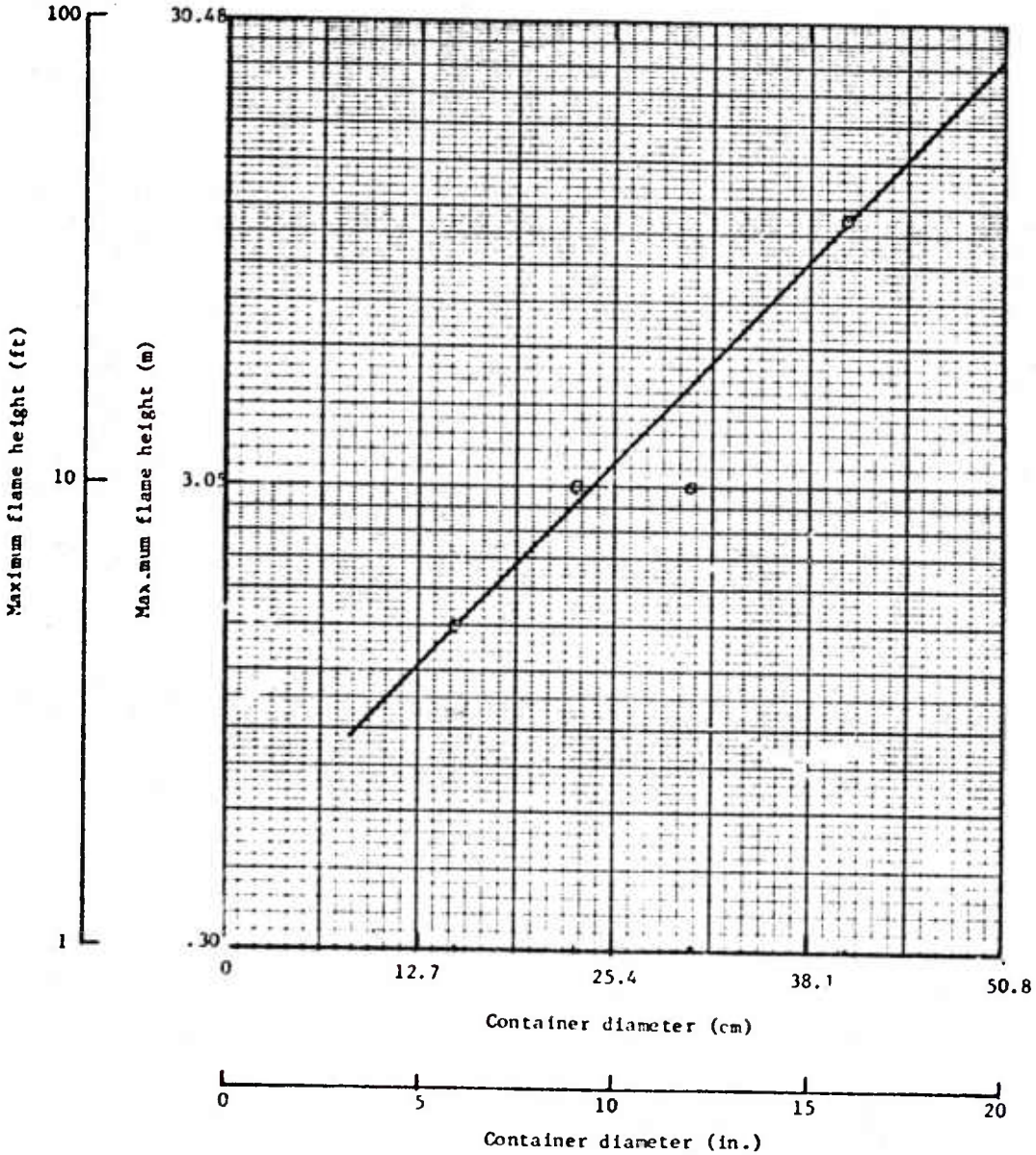

Fig 57 Maximum flame height versus container diameter

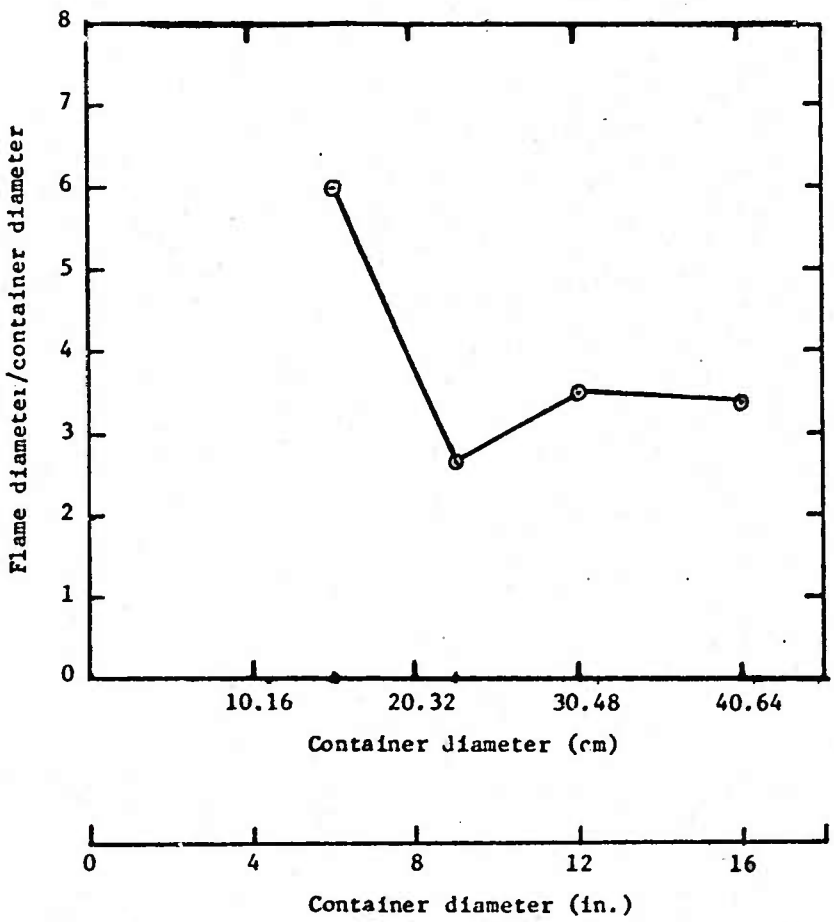

Fig 58 Scaling of flame diameter with container diameter

Test No.	Calculated L/D	Observed L/D
BIB-1	1.11	1.05
BIB-2	4.1	4.33
BIB-3	2.43	2.59
BIB-4	7.96	8.43

All the parameters required for scaling by approach #2 appear to be well controlled, particularly as the container size is increased. Therefore, approach 2 is the primary technique proposed for use in the hazards classification procedure. In the hazards classification procedure, this technique is simplified somewhat by combining parameters. The user will be required to estimate an effective flame emissive power $E_f = \varepsilon_f \cdot \sigma(T_f^4 - T_a^4)$. By plotting E_f versus the test vessel size, scaling can be obtained without going into details such as equivalent flame temperature or absorption coefficient. E_f is estimated for the full scale by extrapolating from the experimental curve. This value is then merely multiplied by the configuration factor F determined from flame height and width as discussed above.

Firespread Test

Purpose

In an automated plant for the manufacture of explosive and/or propellant substances, materials in various stages of the manufacturing process are frequently moved on conveyors from one work station to another within a building, or between buildings. In case an accidental fire should start on one of these conveyors, it is obviously important to extinguish the fire before it propagates to the next work station or the next building. Means are available for sensing the presence of fire and for controlling it, but all require some time for detection and activation. The time required to control a fire must be shorter than the time it would take the fire to propagate from one sensitive location to the next. In fact, the potential rate of fire spread along a conveyor would dictate the minimum safe conveyor lengths between work stations and/or between buildings. That is the conveyor lengths must be sufficiently great so a fire on a conveyor can be controlled before it reaches the next sensitive location. The rates of flame spread along conveyors in various stages of the manufacturing process are therefore critical factors in the layout of the plant facilities.

Current State of the Art

Rates of flame spread differ widely for different combustibles; and for the inprocess materials considered here, they may range from centimeters per min. to hundreds of meter per sec. Furthermore, the rate of flame spread is not a unique physical property of a substance, but depends on many variables that might affect the rate of energy release by the burning material, and the manner in which the energy is dissipated. Such variables may include, for example, the thickness of the fuel layer, its geometric configuration (e.g., flakes, pellets, powders), whether or

not a conveyor is covered and the distance between the fuel surface and the cover, the direction of flame travel (horizontal or at a steep angle), air movement over the surface (drying conveyor), etc.

Unfortunately there are no analytical means for predicting the rate of flame spread for a material based on its physical and/or chemical properties. Nor are there known scaling laws that permit using small bench-type tests for predicting the rate of flame spread for a full-scale condition of actual use. This is true not only for the types of material considered here but also for ordinary wall-finish materials for buildings. For such ordinary materials, test methods have been devised that rank various materials relative to one another, but the results of these tests cannot be used to predict the actual time for fire to spread a given distance on a wall in a building. Tests of this type are not adequate for the present purpose since only real times of flame spread are essential in the layout of a plant for establishing safe distances between sensitive work stations. Thus, with the present state of the art, only full-scale tests on a conveyor mockup could provide the necessary information. Furthermore, in view of the nonuniform (erratic) behavior of explosive and propellant materials, several repetitive tests would be necessary to obtain reliable information with a specified margin of safety. In the conduct of full-scale tests, sufficient data should be collected, even beyond the immediate need, to provide information for future analysis that might lead to reliable modeling and scaling laws.

Nevertheless, there is a need for small-scale testing to guide the design of the full-scale mockups since the latter will be much more costly. The rates of flame spread of different inprocess materials may range over several orders of magnitude, and without some prior knowledge of a material's behavior, the necessary length of a full-scale mockup could not be estimated realistically.

In the hazards classification procedure, it is conceivable that a small or intermediate scale test can be used to estimate flame spread reate on the condition that the estimate is certain to be conservative. If the conservative flame spread estimate puts the user into an economically unacceptable quantity-distance class and if the user has reason to believe that his inprocess material will not propagate fire that quickly in the full scale, an experiment can be done to disprove the result obtained from the smaller scale tests.

Experimental Evaluation

Since M30 pellets in a layer have been shown to be primarily a fire-spread (rather than a mass explosion) hazard in the "critical depth" and "critical length in a layer" tests, firespread tests were required for M30 pellets. Firespread tests are not required for any of the other three sample materials being tested because the other materials do not exist in a layer in the actual process operations.

In firespread tests, celotex troughs (240 cm (8 ft) long) were selected as a standard. In the hazards classification procedure, the process operation's actual layer width and depth should be chosen for the testing. Also, actual materials of construction should be used if they are known. In our evaluation of the firespread test, we varied the trough width at two sizes -- 15 cm (6 in.) and 30 cm (12 in.) and layer depth two sizes -- 1.27 cm (1/2 in.) and 2.54 cm (1 in.) to determine the sensitivity of results to variations in these parameters.

The firespread test is designed to evaluate the damage potential by two mechanisms: (1) by firespread (i.e., is the flame front velocity too fast for the deluge system to respond in time?) and (2) by remote radiant heating from the flame (only if the layer is not covered). If the actual system is covered, the test should also be done with a cover over the layer since this is likely to enhance the firespread. If the actual layer is not covered, flame radiant heating is a potential hazard and should be evaluated in the test. In order to be able to evaluate both firespread velocity and remote heating, we conducted our series of tests in the uncovered configuration.

The arrangement is shown in Figure 59. The layer of pellets was initiated at the far end in the photograph using a gas burner. Radiometers measured heat flux. Movies of the end and side views gave flame shape and flame front velocity. Flame front velocity was also measured using light sensors, seen in Figure 59 at every foot of the trough's length.

The flame front velocity data for the six firespread tests are plotted in Figure 60. Tests FS-1 and FS-2 were both in 1.27 cm (1/2 in.) deep, 15 cm (6 in.) wide troughs. FS-1 used an old batch of M30 pellets with much of the solvent evaporated. Test FS-5 was the same as FS-2 except the trough was 30 cm (12 in.) wide. These three tests gave similar velocity profiles (0.03 m/S). There is apparently some enhancement due to a higher solvent concentration (as would be expected) and some enhancement due to increasing the trough width, but these effects are not extremely strong. In all three cases, the velocity remained fairly constant for the length of the trough, so extrapolation to the full scale length would not be a problem.

Tests FS-3 and FS-4 were both in a 2.54 cm (1 in.) deep layer, 15 cm (6 in.) wide. Good repeatability was seen for these two tests. The data for FS-3 and FS-4 can be fit to a concave upward curve of the form shown in Figure 61 in order to extrapolate to longer trough lengths. The flame travel time for the full scale system t, can be computed from the expression

$$t = \int_0^{\bar{X}} \frac{dx}{V(x)}$$

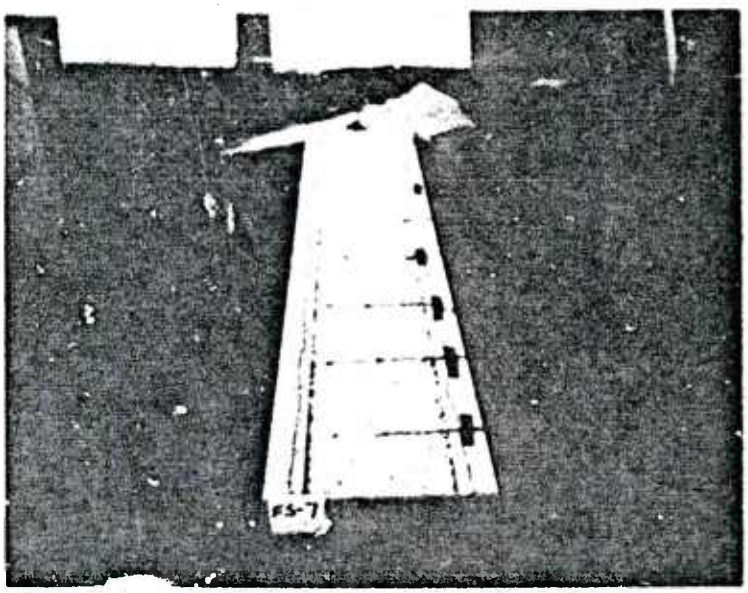

Fig 59 Firespread test arrangement

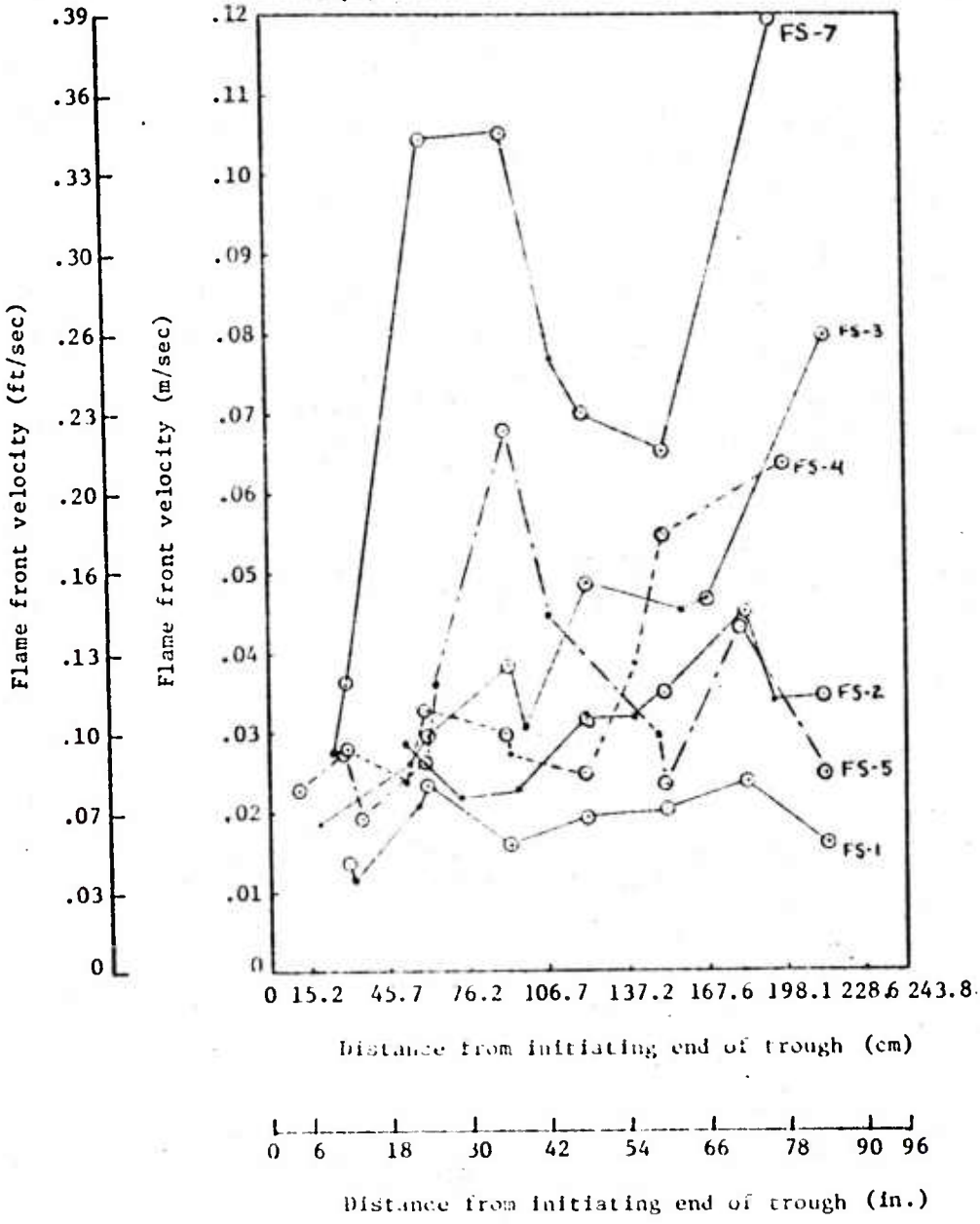

Fig 60 Flame front velocity for firespread tests

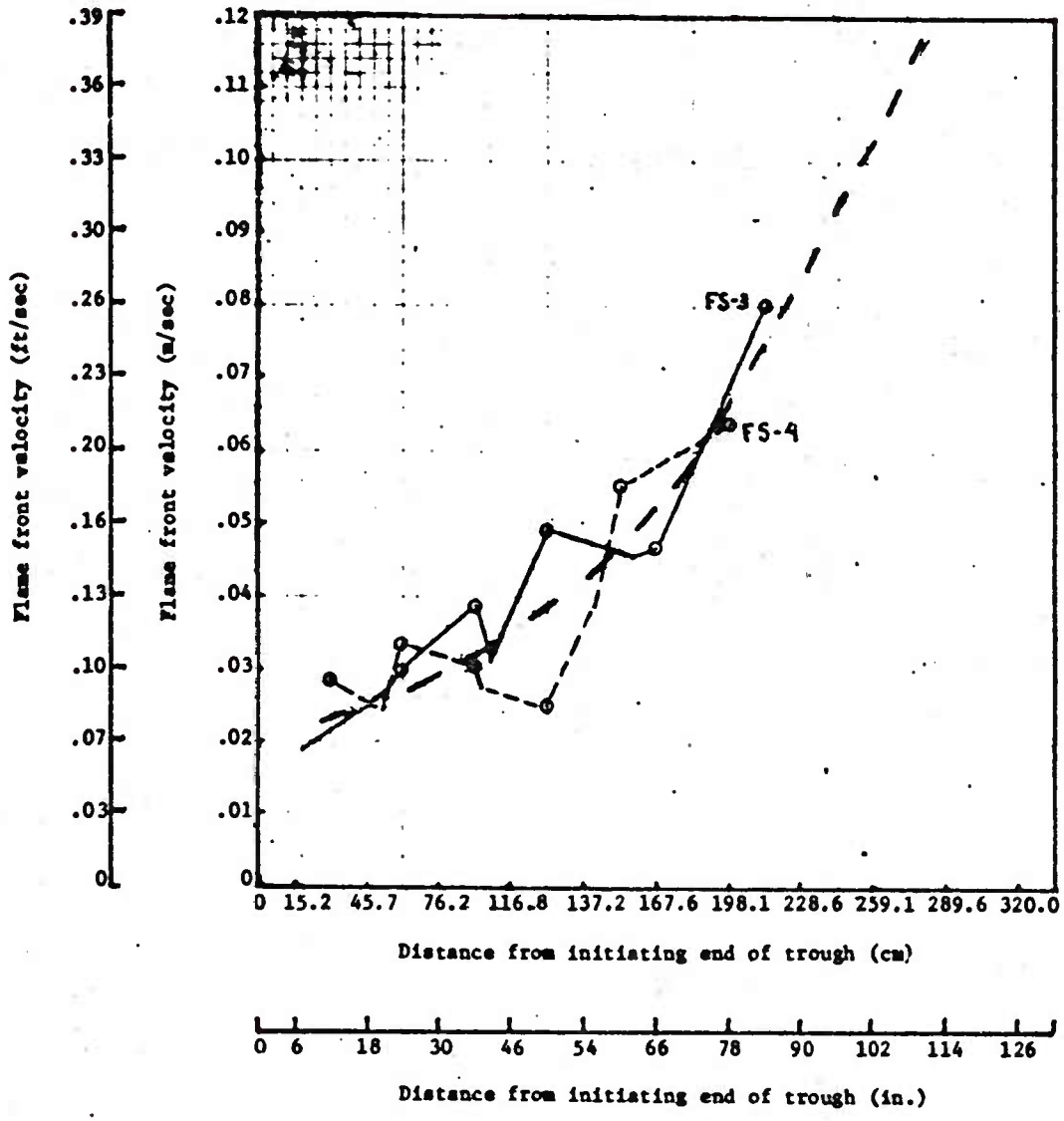

Fig 61 Scaling increase trough length

Where $\bar{X}$ is the full scale trough length, x is length, and V(x) is the flame front velocity versus distance relation. If τ is found to be greater than the total response time of the fire detection/extinguishment system to be used in the plant, either the trough should be made longer or the response time of the detection/extinguishment system should be improved.

In Figure 60, test FS-7 (30 cm wide by 2.54 cm deep) was found to have a higher velocity profile than the other configurations. The velocity profile for this single test was not well defined. If the actual system were 2.54 cm (1 in.) deep by 30 cm (12 in.) wide, several repeat tests should be done to better define the curve shape in order to extrapolate to the full scale length.

While in general, the flame front velocity was found to increase strongly with travel distance, the flame size appears to stabilize after a short distance, although it is somewhat erratic. Flame width and height are plotted in Figure 62 for the tests where movie film coverage of the side view was available. Flame thickness was obtained from the end view movies. Flame shape is strongly related to the remote radiant heat flux, and although the data is somewhat erratic, flame shape can be used to define the maximum remote heat flux produced in a full scale fire.

The heat flux from a flame, $\dot{q}$, can be estimated from the following expression:

$$\dot{q} = \underbrace{(1 - e^{-\alpha x})}_{\varepsilon_f} \cdot \underbrace{F \cos\theta}_{F'} \cdot \sigma \cdot (T_f^4 - T_a^4)$$

Where ε_f is the effective flame emissivity, α is the absorption coefficient, x is the flame thickness, F' is the configuration factor for radiative heat transfer from the flame to the target, F is the configuration factor for a target directly facing the flame and cos θ accounts for the normal to the target's surface being offset by an angle θ, σ is the Stefan-Boltzmann constant, T_f is the flame temperature and T_a is the ambient temperature. The shape factor is given by relations developed by NACA and presented in NACA TN 2836 (see Figure 52). In Figure 52, W is the flame width, h is the flame height and d is the distance from the flame to the target.

Flame temperature can be roughly estimated from the flame color seen in the movies. Based on the yellow color seen in the movies, flame temperature would be expected to be on the order of 930°C (1700°F) to 1040°C (1900°F). To estimate α, we chose to use test FS-7 and 1040°C (1900°F) as a standard. This yielded an α of 0.83/cm (0.252/ft), which is reasonable for flames of this type. By using this value and rearranging the expression for heat flux, the effective flame temperature T_f could be computed for each test where heat flux and flame size data were available.

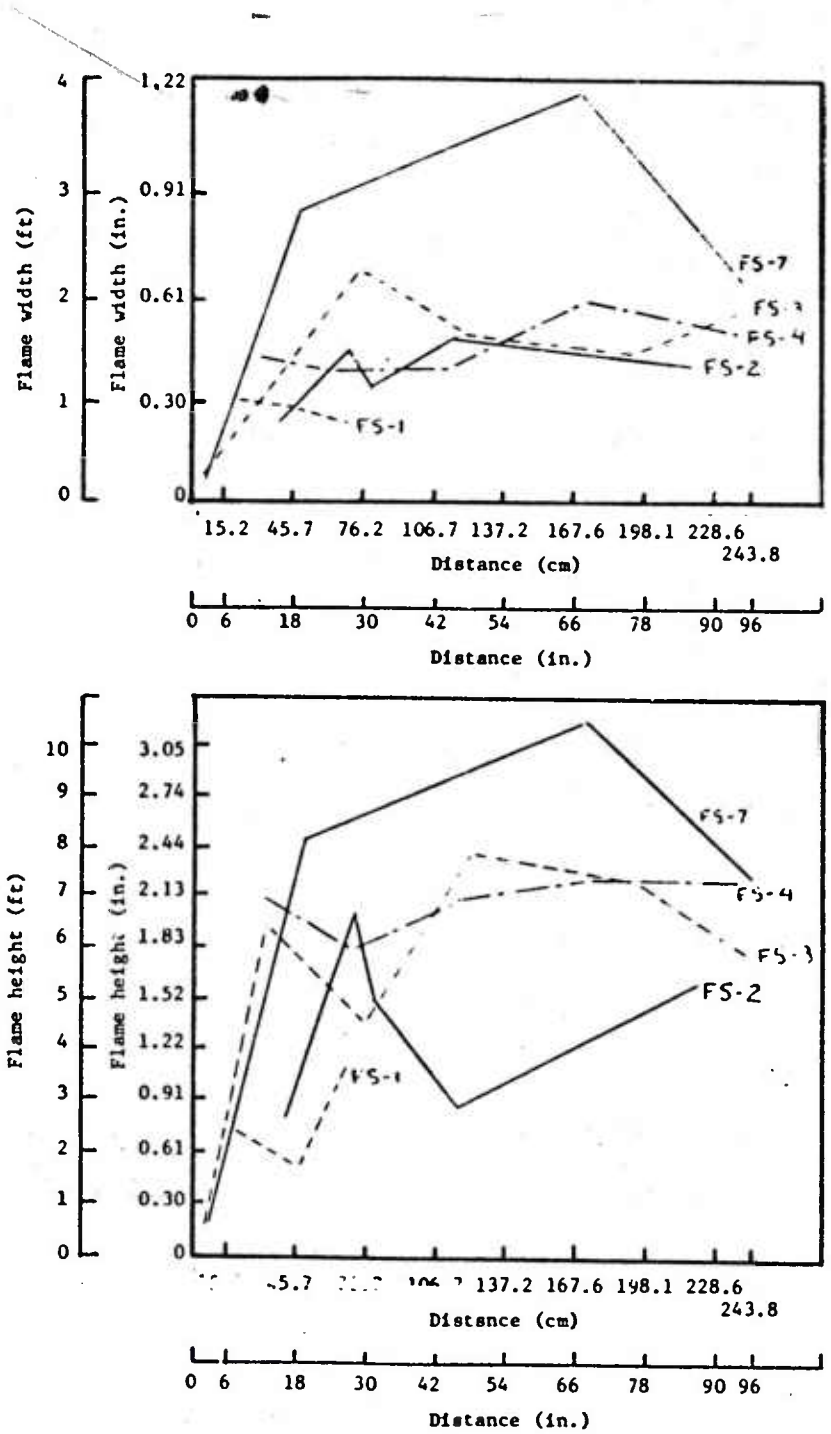

Fig 62 Characteristic flame dimensions versus flame front, distance

The flame temperature computations are presented in Table 25. In all cases except test FS-1, the flame temperature was right in the range 930°C (1700°F) to 1040°C (1900°F). Test FS-1 used an old batch M30 pellets with most of the solvent evaporated. The other tests used fresh sample. It appears that the effective flame temperature can be assumed to be a constant for a given sample material. Once the effective flame temperature is known, scaling can be accomplished purely based on the flame shape using the heat flux equation given above. Based on the fire-spread test results, a conservative flame temperature of 1040°C (1900°F) can be used and flame shape can be inferred from Figure 50 in order to estimate the heat flux which would be produced from a fire in a full length trough.

Cloud Explosion Test

In process operations, there are many systems in which a fine powder or a vapor is dispersed in air inside of a container. These systems include pneumatic mills (e.g., jet mill), pneumatic mixers, cyclone separators, pneumatic ducts, etc. For these systems, the process material which presents a potential hazard is the chemical dispersed in air rather than the chemical by itself. To characterize the potential explosion hazard for such a system, the Hartmann dust explosibility apparatus (Ref 22) or the Bartknect apparatus (Ref 23) can be used. The Hartmann apparatus is suggested for use in the hazard classification procedure at the present time because the test method is well established and the apparatus exists at many facilities. It has been shown that the Hartmann apparatus is too small for scaling the pressure rise characteristics of a cloud explosion (Ref 23), while the Bartknect apparatus is sufficiently large. In addition, The Bartknect apparatus can test vapors as well as dusts. The Hartmann is only for dusts. Therefore, tests done in a Bartknect apparatus are atleast as good as Hartmann results and always acceptable. However, since the Bartknect type test requires a large costly chamber and the equipment is presently only available at about three laboratories, the Hartmann test will be used for the present.

In the hazard classification procedure, only a general test description is provided. The ASTM E-27 Dust Subcommittee has a detailed draft of a Standard Dust explosion test using the Hartmann apparatus. The draft is still not ready to be released and is therefore not incorporated in this report. However, it is suggested that the final ASTM Standard for this test be adopted for hazards classification when the standard is finalized.

Table 25

Computation of effective flame temperature

Test	Flame position	Measured heat flux		Flame height		Flame width		X[a]	Y[a]	F'[a]	Flame thickness		ϵ_f	T_f	
		w/m^2	$(btu/ft^2\text{-}sec)$	m	(ft)	m	(ft)				m	(ft)		°C	°F
FS-1	End of second third	227	(0,02)	0.76	(2.5)	0.27	(0.9)	0.50	0.09	0.0256	0.35	(1.16)	0.254	2082	(1139)
FS-2	End of second third	2610	(0.23)	1.22	(4.0)	0.46	(1.5)	0.80	0.15	0.056	0.42	(1.37)	0.292	3403	(1873)
FS-2	End of trough	3110	(0.274)	1.62	(5.3)	0.40	(1.3)	0.88	0.217	0.067	0.42	(1.37)	0.292	3398	(1870)
FS-4	End of second third	3972	(0.35)	2.26	(7.4)	0.55	(1.8)	1.48	0.18	0.08	0.64	(2.1)	0.411	3121	(1716)
FS-4	End of trough	5913	(0.521)	2.23	(7.3)	0.52	(1.7)	1.22	0.283	0.083	0.94	(3.1)	0.542	3204	(1762)
FS-7	End of second third	10,373	(0.914)	3.05	(10.0)	1.16	(3.8)	2.00	0.38	0.17	0.55	(1.8)	0.365	3452	(1900)

[a] F' is computed from Figure 53 including the effect of the target orientation angle θ. The parameters X and Y are those defined in Figure 53

HAZARDS CLASSIFICATION PROCEDURE

The hazards classification procedure developed under this program for inprocess propellant and explosive materials in given in Appendix E. The procedure is the outgrowth of all the investigations described in the previous sections of this report. The procedure consists of a general background section (Chapter 1), a description of the overall procedure steps (Chapter 2) and an appendix containing detailed procedures for each of the individual tests which may be required. It should be noted that not all of the tests described will be required for any given material to be classified.

The overall procedure consists of a sensitivity evaluation (sensitivity tests) and an effects evaluation (screening tests and effects tests). The sensitivity evaluation leads to a sensitivity classification for the material (either "sensitive" or "insensitive"). The sensitivity classification is based on a comparison of sensitivity test results for stimuli which could occur in the actual process operation to "high credible" stimulus energy levels for the specific process being considered. The "high credible" values are based on an analysis of historical process plant accident data and simple hazards analysis calculations. The values obtained by these methods may be extremely rough approximations and/or quite questionable in some cases. A complete additional program could be devoted to estimating the best stimulus energy values to use for each type of process operation. This probably would not be warranted in that the sensitivity classification is only meant to be an approximate qualitative indication of how likely an ignition is to occur and which stimuli will be the most likely causes. This information is useful for safe handling of the material, but not as important as knowing the ultimate result of an initiation. The major emphasis in this hazards classification procedure lies in the effects evaluation. The effects evaluation characterizes the material in terms of the most likely consequence of an initiation. The NATO-UN categories (Ref 3) have been expanded slightly to adopt them better to inprocess situations. As the result of the effects evaluation, the material is placed into one of the modified NATO-UN classes.

When developing the effects evaluation procedure several logical options existed in terms of using the critical size and transition tests to screen the sample and choose the most appropriate effects test to be done. In determining the best logic scheme, we had four screening tests and four effect tests to work with. These are

Screening Tests

1. Critical Diameter: Can the material sustain an existing detonation in its process vessel?
2. Tube Transition: Can a detonation develop from a flame in the process vessel?

3. Critical Layer Thickness: Can the material sustain an existing detonation in the process layer depth?

4. Layer Transition: Can a detonation develop from a flame in the material at the process layer depth?

Effects Tests

1. Mass Explosion Test: Characterizes explosive airblast and fireball

2. Cloud Explosion Test: Characterizes severity of a dust or vapor explosion in the process vessel

3. Mass Fire Test: Characterizes the potential for the spread of fire and damage from a mass fire of the material in bulk

4. Firespread Test: Characterizes the fire severity and flame spread rate for materials in layers

The first possibility was to omit the screening tests and begin with the most severe effects test. For example, always conduct the mass explosion test first (except for dust or vapor clouds). If the material is not classified as a mass explosion hazard by the test, then go to the next lower effects test, the mass fire test, and so on. When testing this approach using the test results for the four sample materials evaluated, it was found that the sample would always be classified conservatively (all four materials were class 1.1A, mass explosion hazard). This result was felt to be unreasonably stringent in that in a real accident some of the materials clearly would pose a fire hazard and not an explosion hazard.

The second approach was to use only the critical dimension tests (ask, is the material detonable?) and not use the transition tests to help select the most appropriate effects test to be done. In using this approach, we are not concerned with whether the detonation develops in the particular process vessel being studied. Rather, detonation could develop elsewhere and the sample being tested must only be able to propagate the detonation in order to be classified 1.1A. This approach is probably the most realistic conceptually however, when trying it using the test results for the four sample materials, the approach was found again to be overly conservative.

The third approach which was tried adds the transition tests to help in screening. This approach assumes that initiation must originate and build to a detonation in the process vessel being evaluated, in order for the sample to be classified 1.1A. By adding the transition tests to the screening, the most reasonable results were obtained for the four sample materials tested. The judgement of what was reasonable was based on observation of the effects tests and consideration of the actual process configurations. None of the approaches embody perfect logic for routing the evaluation to the most proper effects test, but the third approach seems to embody the best balance of optimisms and conservatisms to generally give the most realistic answers.

PROCEDURE VALIDATION AND CONCLUSIONS

In order to assure that the overall procedure logic is reasonable and that the required test procedures do not embody unforseen problems, the hazards classification procedure has been applied to the four sample materials selected in the previous project.

The four sample materials used in this program were quite old when testing was done and do not necessarily accurately represent the actual inprocess materials. Therefore, we were not accurately classifying the four materials here. They were used only to test out the validity of the procedures being proposed. In order to test the overall hazards classification procedure using these samples, the following assumptions were made concerning the process operations from which the samples were extracted:

1. The RDX slurry is assumed to be from a conveying operation most similar to a chute. It is assumed to exist in the nearly dry form as it was received, rather than as 15 percent solids in water.
2. M30 pellets are assumed to exist inprocess on a belt conveyor dryer which is 6 m long by 0.3 m wide by 1.27 cm deep. The belt is assumed to be inside a large 2.4 m (8 ft) tall oven with other conveyors. The material on the other conveyors could be ignited if the original fire were severe enough.
3. M26 paste is assumed to exist inside a 0.9 m diameter by 1.52 m long vessel.
4. After extrusion, the M1 strands are assumed to be piled into a container which is 0.3 m diameter by 0.61 m long. The room's ceiling height is 6.1 m, or about 5.5 m above the top of the container.
5. All process vessel walls are assumed to be 0.32 cm (1/8 in.) thick.

These assumptions may or may not be correct for the actual system. However, since we aren't really concerned with accurately classifying the materials, these assumptions will serve well to test out the procedure.

The results of the classification of the four materials are summarized in Tables 26 through 29. The procedure seems to work reasonably well. No problems were encountered in applying the system to the four sample materials tested. A qualitative description of the likelihood of initiation and most probable stimuli is provided as well as what seems to be a realistic effects classification. Whether or not the procedure realistically classifies all inprocess materials needs much more extensive validation. A more thorough validation of the procedure may indicate that some of the specific criteria values need to be shifted somewhat or that some aspect of the overall logic needs adjustment.

Table 26

Classification of RDX slurry[a]

Sensitivity evaluation

Test required	Inprocess potential	Sample sensitivity	Safety factor
Local impact	5.3×10^4 j/m^2	1.9×10^6 j/m^2	36
Impingement	10 m/sec	180 m/sec (Ref 19)	18
Rubbing friction	4.9×10^8 w/m^2	$>4.65 \times 10^8$ w/m^2	>0.95
ESD charging susceptibility	(Marginal--relaxation time = 25 msec)		
ESD ignition	0.17 j	> 1 j	5.88
Thermal (autoignition)	100°C	255°C	2.55
Thermal (local hot spot)	1000°C	> 1066	> 1.066
		System safety factor due to thermal stimuli	> 1.066

Effects evaluation

(1) Critical diameter was found to be < 0.64 cm.
(This is less than the process vessel diameter, therefore the tube transition test is required.)

(2) Critical length was found to be negligible.
(This is less than the process vessel length, therefore the mass explosion test is required.)

(3) The TNT equivalency for the RDX sample was found to be about 120%.
(This is greater than 10%, therefore the material is Class 1.1A, mass explosion hazard.)

Classifications

Class 1.1A (mass explosion hazard)
and
SENSITIVE due to thermal stimuli

[a]Note, tests were done with the RDX as received. The as received material required the addition of a significant quantity of water to realistically simulate the actual inprocess material form.

Table 27

Classification of M26 paste

Sensitivity evaluation

Test required	Inprocess potential	Sample sensitivity	Safety factor
Local impact	5.2×10^4 j/m^2	6.92×10^5 j/m^2	13
Rubbing friction	6.5×10^9 w/m^2	$> 7.20 \times 10^8$ w/m^2	> 1.1
ESD charging susceptibility	(Not susceptible--relaxation time = 0.2 msec)		
ESD ignition	0.17 j	1 j	5.88
Thermal (autoignition)	124°C	183°C (assumed value)	1.48
Thermal (local hot spot)	1000°C	500°C	0.5

System safety factor = 0.5
due to thermal stimuli

Effects evaluation

(1) Critical diameter was found to be about 0.9 cm.
(This is significantly smaller than the process vessel diameter, therefore the tube transition test is required.)

(2) Critical length was found to be about 9.14 cm.
(This is significantly shorter than the process vessel length, therefore the mass explosion test is required.)

(3) The TNT equivalency of M26 paste was found to be between 80 and 130%. (This is greater than 10%, therefore this material is Class 1.1A, mass explosion hazard.)

Classifications

Class 1.1A (mass explosion hazard)
and
SENSITIVE due to thermal stimuli

Table 28

Classification of Mi strands

Sensitivity evaluation

Test required	Inprocess potential	Sample sensitivity	Safety factor
Local impact	6.76×10^4 j/m^2	$> 3.14 \times 10^6$ j/m^2	> 46
Rubbing friction	4.90×10^8 w/m^2	$> 1.48 \times 10^8$ w/m^2	> 0.3
ESD charging susceptibility	(Highly susceptible to changing--relaxation time =		
ESD ignition	0.17 j	> 1 j	> 5.88
Thermal (autoignition)	340°C	118°C (Ref 19)	0.347
Thermal (local hot spot)	1000°C	786°C	0.786
		System safety factor = due to thermal stimuli	0.347

Effects evaluation

(1) Critical diameter was found to be about 1.9 cm.
(This is smaller than the process vessel diameter, therefore the tube transition test is required.)

(2) Critical length was found to be greater than 1.37 m.
(This is larger than the process vessel length, therefore the mass fire test is required.)

(3) The mass fire test indicated that the flame could extend 4.88 m high and give a heat flux of 0.87 w/cm^2 at 3 m from the flame.
(At the indicated flame height, the flame tip is more than 10% below the ceiling but the flame's heat flux at 3 m is greater than the criteria of 0.728 w/cm^2. Therefore, the material is class 1.3A.)

Classification

Class 1.3A (mass fire hazard)
and
SENSITIVE due to thermal stimuli

Table 29

Classification of M30 pellets

Sensitivity evaluation

Test required	Inprocess potential	Sample sensitivity	Safety factor
Local impact	5.3×10^4 j/m^2	$> 4.45 \times 10^5$ j/m^2	> 8.5
Impingement	10 m/sec	63 m/sec (Ref 19)	6.3
Rubbing friction	4.9×10^8 w/m^2	$> 1.23 \times 10^8$ w/m^2	> 0.25
ESD changing susceptibility	(Not susceptible--relaxation time = 1.3 msec)		
ESD ignition	0.17 j	> 1 j	> 5.88
Thermal (autoignition)	481°C	170°C	0.35
Thermal (local hot spot)	1000°C	1035	1.035
		System safety factor = due to thermal stimuli	0.35

Effects evaluation

(1) Critical layer thickness was found to be about 7.6 cm. (This is thicker than the process layer depth, therefore, the firespread test is required.)

(2) The flame spread test showed the maximum flame height to be about 2.3 m, the maximum heat flux at 3 m to be about 0.24 w/cm^2, and the estimated flame travel time to be about 174 sec across a 6.1 m long layer. (The enclosure is assumed to be 2.47 m tall and the flame height is within 10% of this value, therefore, the material is class 1.3A, mass fire hazard. Note, that this is a borderline case. If the flame were slightly shorter, the heat flux and flame spread time are both well on the safe side of their criteria and the material would have been class 1.4, minor hazard.)

Classification

Class 1.3A (mass fire hazard)
and
SENSITIVE due to thermal stimuli

RECOMMENDATIONS

For these reasons, it is recommended that a much more extensive validation of the procedure be conducted. Materials with known accident histories (good as well as bad) should be used so that a clear indication can be obtained whether or not the procedure conclusions are realistic.

It is also recommended that a program be devoted to assuring that the required sensitivity tests for each process operation as well as the inprocess potential energies for each case are realistic. It is suggested that each case (that is each stimulus for each process operation) be looked at in detail to assure that the inprocess energy levels with which the sensitivity test results are to be compared are indeed realistic "high credible" values. It should be kept in mind that the primary classification is based on the effects of an initiation rather than on the material's sensitivity. However, to assure that the sensitivity evaluation is meaningful (even qualitatively) the required tests for each process operation and the inprocess potential energies should be carefully scrutinized and selected with confidence.

Several of the proposed test methods could still be improved somewhat with additional work. For example, the rotary friction apparatus used on this program was made up of available components and used a drill press structure as the framework. The apparatus was quite sturdy and worked reasonably well, however, a special design for the equipment using optimum components should provide a substantial improvement. In the ESD ignition test, more research into the relation between spark gap, capacitance, spark energy and electrode configuration may lead to a reduction in the number of tests required. The tube transition test may also be improved with better understanding from additional testing. The test is the final stage of an evolution process which occurred during the program and only one trial was completed for each sample in that configuration.

Finally, it is felt that the proposed hazards classification procedure is representative of the current state of the art. Since our understanding of the various phenomena is continually advancing, it is recommended that the procedure be reevaluated periodically (e.g., once every five years) and modified to reflect advances in the state of the art. It is also suggested that a comprehensive effort be directed toward defining criteria for safe handling and safe separation of the materials in each class. A substantial base already exists in this area but emphasis has been on mass explosions involving materials in storage or transport. Work is needed for inprocess materials, particularly in defining safe handling and safe separation critieria for the effects other than mass explosion.

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APPENDIX A

SUMMARY OF ACCIDENT REPORTS FROM DDESB FILE

SUMMARY OF SELECTED ACCIDENTS WHICH OCCURRED DURING MELT-POUR CASTING

ASESB NO	AGENT	AMOUNT (LB)	FATALITIES/ INJURIES	COMPONENT OR OPERATION	OUTPUT-TYPE	MAX. DISTANCE MISSILE/GLASS BREAKAGE (FT.)	PROBABLE CAUSES
395	Amatol 60/40	150	0/0	Melting Process-cascade	Fire		Spontaneous ignition at lower sieve-impurities-thermal
433	15% Aluminum, 15% Hexogen 70% TNT	18.000 + 116.000		Melt-pour Facility	Explosion	300/-	1) Thermal: improper temperaure control of kettle contents
8	Sealing wax. H.E. antiair-craft projec-tiles		0/2	Melting Operation	Fire-Explosion		Unknown, no specification
41	Molten TNT	11,000-14,000	9/16	Dopp Kettle Feeder Fa 2000 lb bombs	Explosion		Unknown
116	TNT	110,000	8/1	Casting/cooling Shed	Explosion		1) Friction 2) Impact
140	Molten TNT (81 mm mortar)	2000	13/27	Melt-pour Operation	Explosion		1) Impact (or TNT dust) 2) Dust or vapor ignition inside melt hood-thermal
141	TNT Melt		22/84	Continuous Melt Unit	Explosion	300-400/-	1) Friction 2) Pinching
191A	Amatol Mixture 50/50 (TNT-Ammonia Nitrate)		66/23	Pouring molten TNT into shells	Explosion		Unknown
235	TNT	10		Melt kettle	Fire		Thermal-steam line to kettle overheated
297	TNT 1. crude 2. processed 3. finished	28 tons 28 tons 28 tons	75/119	Melting	Fire-Explosion		Unknown, no specification
813	Experimental propellant		1/1	Casting pro-pellant	Explosion-Fire		Friction-propellant shaft pocking system inade-quately designed
1099	Cyclotol 70% RDX, 30% TNT		2/?	Melt Pour Operation	Explosion		Unknown, not legible
1122	Lead Styphnate	2 kg	1/0	Pouring Operation	Explosion		Unknown, no specification
1332	Cyclotol	4000-5000	0/1	Melt Pour Building	Explosion		Unknown, no specification

SUMMARY OF SELECTED ACCIDENTS WHICH OCCURRED DURING MELT-POUR CASTING (con't)

ASESB NO.	AGENT	AMOUNT (LB)	FATALITIES/ INJURIES	COMPONENT OR OPERATION	OUTPUT-TYPE	MAX DISTANCE MISSILE/GLASS BREAKAGE (FT)	PROBABLE CAUSES
386-19	Picric Acid			Melt Pot	Fire		Spontaneous ignition of vapor-thermal (?)
386-20	Picric Acid			Melt Pot	Fire		Spontaneous ignition of vapor-thermal (?)
386-21	Picric			Melt Pot	Fire		Spontaneous ignition of vapor-thermal (?)
1275	TNT		0/0	Melt Tank	Fire		Friction/spark initiation during removal of old (TNT) contaminated insulation (maintenance)
518	RDX/TNT (6) torpedoes		11/	Melting Process	1) Bomb 2) Fire-Explosion	500 yrd proximity completely destroyed	Foreign material inclusion, cardboard soaked with oil caught fire during melting (fuel & thermal)
601	Lead Azide	1) 10 gr detonator	0/1	Unloading molds from extraction unit	Explosion	5-10'/--	Invested mold was brought in contact with surplus explosive on top of the extraction machine
906	TNT	Residual TNT contaminate	0/0	Melt reservoir	Fire	NA	Impact of contaminated bottom of tank, residual TNT on bottom exposed to 200°F for 9 hours
1066	1) 4-Polaris A3 second stage	--	3/11	Prop. motor casting	Explosion	1500/2000	Solvent handling - vapor initiation
	2) Nitroglycerine	4800					
	3) Casting powder	865					
	4) Scrap casting solvent	250					
	5) Aspirator	30					
989	Pentolite (50/50)	/20	0/0	Melt tank	Explosion & Fire	100/0	Instability of Pentolite under prolonged heat

SUMMARY OF SELECTED ACCIDENTS WHICH OCCURRED DURING MELT-POUR CASTING (concl)

ASESB NO.	AGENT	AMOUNT (LB)	FATALITIES/ INJURIES	COMPONENT OR OPERATION	OUTPUT-TYPE	MAX. DISTANCE MISSILE/GLASS BREAKAGE	PROBABLE CAUSES
868	TNT	Residual TNT on floor	0/1	Melt chamber	Fire		Friction initiation due to scraping of dry TNT residual on concrete floor with steel spatula
1230	Cyclotol		6/4	Melt and pour operation	Explosion-Fire		1) Spark initiated - tool droppage 2) Riser scrap causing friction between agitator and kettle 3) Foreign material present-friction 4) Contamination of electrical controls with explosive dust, etc.

SUMMARY OF SELECTED ACCIDENTS WHICH OCCURRED IN TOTE BINS

ASESB NO.	AGENT	AMOUNT (LB)	FATALITIES/ INJURIES	COMPONENT OR OPERATION	OUTPUT-TYPE	MAX. DISTANCE MISSILE/GLASS BREAKAGE (FT.)	PROBABLE CAUSES
1225	RD 13-33 NOL Prime 130 Dextrolead Azide	105	2/0	Service Supply Powder Buggy	Explosion	483/	Impact-dropped NOL 130 into buggy

SUMMARY OF SELECTED ACCIDENTS WHICH OCCURRED IN HOPPERS

ASESB NO	AGENT	AMOUNT (LB)	FATALITIES/ INJURIES	COMPONENT OR OPERATION	OUTPUT-TYPE	MAX DISTANCE MISSILE/GLASS BREAKAGE (FT)	PROBABLE CAUSE
359	Not specified	500	5/0	Hopper of Screw Filling Machine	Explosion		Friction (foreign object in hopper)
1273	Gilsonite, sulfur, aluminum flies		0/2	Charging/Loading Feed Hopper	Fire		Unknown
364	Amatol 80/20	75	3/1	Hopper/Screw Filling Operation for Extrusion	Explosion	300/-	Friction (foreign object or solid sensitized Amatol in feed)
608	5 grain A-Z Detonators		0/2	Delivery Chute of Filling Machine	Explosion		Friction from tapping disrupting blockage in chute
636	6 grain ZY detonator (Lead Azide and CE)		0/0	Feed Hopper of Filling Machine			Friction between guide plate and charge plate
1547	C-4			Hopper Dump Operation	Explosion		Unknown-no specification
1598	Primer		0/0	Syntron Primer Hopper Dump Phase	Explosion		Unknown-no specification
120	Smokeless Powder	4000	1/0	Filling Feed Hopper in Screen House	Fire-Low Order Detonation		Impact-mechanical failure of hopper
234	Smokeless Powder		3/0	Chute for/Solvent Recovery Process	Explosion		1) Friction - during trembling impingement 2) ESD
1168	NIBEX-High Energy Propellant (Zirconium)	150	0/3	Dumping into Hopper	Fire-Explosion		1) Impact 2) ESD
334(e)	Black Powder			Supply Hopper Filling Operation for Pelleting Machine	Explosion		Friction
1296	Multi-perforated single base propellant	1900-7000	2/4	Drop Plug Buggy/ Hopper Removal Proposed	Fire-Explosion		1) ESD 2) Impact

SUMMARY OF SELECTED ACCIDENTS WHICH OCCURRED IN HOPPERS (cont'd)

ASESB NO.	AGENT	AMOUNT (LB)	FATALITIES/ INJURIES	COMPONENT OR OPERATION	OUTPUT-TYPE	MAX. DISTANCE MISSILE/GLASS BREAKAGE (FT)	PROBABLE CAUSE
499	Smokeless Powder 40 mm cannon		3/13	Feed Hopper in Blending Operation	Fire		1) Friction 2) Impact
592	Nitro-glycerine			Hopper/Buggy in Mix House			Unknown-no specification
747	Mercury Fulminate Potassium Chlorate Antimony Sulfide, Sulfur, Sealed Powder		0/1	Filling Hopper Supply	Explosion		Friction
824	Photoflash Composition for 762 mm Rockets		0/0	Hopper Feed Chute	Explosion		1) Impact (foreign body) 2) Friction (from dump gate valve assembly a foreign particle)
1011	Giant Gel 40% Dope, Sulfur		0/0	Feed Hopper/Weigh Station	Flash Ignition		1) Impact 2) Friction hard aluminum and steel interface 3) Air dust mixture-friction
569	Smokeless Powder	130,000	9/9	Filling Bin	Fire		1) Friction-metal-metal contact by opening slide date on car 2) ESD
755	Multi-perforated single base M1 cannon powder and graphite dust	3000 and additional 2000	2/3	Loading pre-blender hopper	Explosion-Fire	900/4500	1) Static discharge-due to powder impingement 2) Potential difference between accumulated static charge of powder in buggy and the powder in metal barrel 3) Potential difference between operator and powder 4) Friction between hopper edge and buggy

SUMMARY OF SELECTED ACCIDENTS WHICH OCCURRED DURING RECRYSTALLIZATION

ASESB NO	AGENT	AMOUNT (LB)	FATALITIES/ INJURIES	COMPONENT OR OPERATION	OUTPUT-TYPE	MAX. DISTANCE MISSILE/GLASS BREAKAGE (FT)	PROBABLE CAUSE
224	RDX	15	0/2	Recrystallization Process	Explosion		1) Impact 2) Hot Spot - Temperature 3) Friction (metal-metal)
3cc *	Picric Acid			Crystallizing Process	Fire		Friction
1222	RDX Slurry		0/1	Valve for re-crystallization still	Explosion		Impact, unplugging of valve with nonsparking screw driver

SUMMARY OF SELECTED ACCIDENTS WHICH OCCURRED DURING NEUTRALIZING

131	Nitro-glycerine	2557	1/9	Neutralizing House	Explosion	-/400	1) Impact 2) ESD
290	Nitro-glycerine	3700	6/0	Neutralizing House/Plug Valve Opening Process	Explosion		1) ESD 2) Electrical 3) Friction/Impact - Mechanical Equipment

SUMMARY OF SELECTED ACCIDENTS WHICH OCCURRED DURING GLAZING

41	Detonite	9000		Glazing Facility	Explosion	1500-5000/ 2 miles	Unknown
90	Black Powder		0/0	Glazing Mill	Explosion	300-600/	Electrical (lighting)
94	Black Powder	875,000	24/34	Glaze-Pack House	Explosion-Fire	Structural Damage to 4,000 ft	Unknown - No specification
741	Ball Powder WC870	1060	1/- (Burn)	Salt coat and glazing, Sweetie barrel	Explosion	Immediate area	Slurry spillage-alcohol fumes ignited by metal-metal contact (bucket and object)
41	Black Powder	5000	0/0	Glazing	Explosion	850 ft/1½ miles	Lightning
1330	1) Semi-finished black powder	7000	1/1	Glazing	Explosion	½ mile/2 miles	Unknown
	2) Finished black powder	1300					

SUMMARY OF SELECTED ACCIDENTS WHICH OCCURRED DURING PUMPING OPERATIONS

ASESB NO	AGENT	AMOUNT (LB)	FATALITIES/ INJURIES	COMPONENT OR OPERATION	OUTPUT-TYPE	MAX DISTANCE MISSILE/GLASS BREAKAGE (FT)	PROBABLE CAUSES
-07	TNT (liquid)	11 000	2/3	Pumping TNT from fortifier	Explosion		1) Friction \ Overheated pump caused 2) Impact / by impeller casing interaction
342-27	Not specified			Piping/continuous melt operation	Explosion		Unknown, no specification
1304	RDX/TNT swarf	3		Vacuum pump	Fire	NA	Pump overheating
1032	HMX slurry		0/0	Pump	Explosion		a) HMX caking within pump cavity caused friction b) Presence of supersensitive Alpha fumes of HMX
1052	DNT/TNT (70-30%)		0/0	"Downie" pump	Fire	NA	1) Localized overheating within pipe casing 2) Foreign objects
1280	Propellant Single base		0/0	Slurry delivery pipeline	Explosion-Fire	250/-	Thermal decomposition due to heat application from steam tracer line and propellant accumulation within pipe

SUMMARY OF SELECTED ACCIDENTS WHICH OCCURRED IN BELT CONVEYORS

ASESB NO.	AGENT	AMOUNT (LB)	FATALITIES/ INJURIES	COMPONENT OR OPERATION	OUTPUT-TYPE	MAX. DISTANCE MISSILE/GLASS BREAKAGE (FT)	PROBABLE CAUSE
625	4.2 in Mortar		0/4	Belt Conveyor loading line during removal of shell	Explosion		Impact on firing pin
1214	M406 40 mm Rd: Comp B and Comp A5	32 g		Belt Conveyor/ Assembly	Explosion		Defective freeze
1302	Primer Electric 52, Lead Styphnate, Graphite Potassium, Chloride Barium Nitrate		0/5	Belt Conveyor loading filling line	Explosion-Fire		Friction
593	Nitroglycerine				Explosion	/750	Friction: wood scraper and compound

SUMMARY OF SELECTED ACCIDENTS WHICH OCCURRED IN SCREW CONVEYOR TYPE

812	Rakrift Explosion (Nitroglycerine and Nitroglycol)	200	4/15	Feeder Extruder Screws	Explosion		1) Impact 2) Friction (metal-mold)

SUMMARY OF SELECTED ACCIDENTS WHICH OCCURRED IN PNEUMATIC CONVEYOR

1314	Commercial Powder	Total 8210 (12,000 lbs/hr)	0/6	Air-conveying of powder for reblender	Explosion	Immediate building 82/-	1) Friction heat buildup of particles inside rubber hose (deterioration of hose) 2) Extreme velocities of transfer

SUMMARY OF SELECTED ACCIDENTS WHICH OCCURRED IN FILTERS

ASESB NO.	AGENT	AMOUNT (LB)	FATALITIES/ INJURIES	COMPONENT OR OPERATION	OUTPUT-TYPE	MAX. DISTANCE MISSILE/GLASS BREAKAGE (FT)	PROBABLE CAUSE
1683	6 amino-penicillanic acid S-oxide (trimeric acetone peroxide exploded)		0/1	Filter/Reactor	Explosion		1) Friction-technician touched filter cake with steel spatula 2) ESD - tests show explosion occurs at 11.5 mJ electric spark
1182	Chemical filter solution Mithanol (50-50) and 5% caustic		0/0	Decontamination/ Cleaning	Explosion/Fire		Fuel-Air Ignition: 1) Friction 2) ESD 3) Heat and reaction thermochemical

SUMMARY OF SELECTED ACCIDENTS WHICH OCCURRED DURING SEPARATION OPERATIONS

460	Nitroglyc-erin	3800	2/1	Nitration Process/ Separator Phase	Explosion	400-600/2 miles	Thermochemical reaction, decomposition of nitroglycerine
263	Di and Tri-Nitrotoluene			Separation process/scooping material residue for further processing (wash)			1) Friction) Dropped article 2) Impact) into container 3) Incompatible material
1109	Nitrated sugar glycerine	400		Separator	Explosion	1920/-	Thermochemical decomposition

SUMMARY OF SELECTED ACCIDENTS WHICH OCCURRED DURING DISTILLATIONS OPERATIONS

1019	Di-nitro fluourethane (Daphne)	20.75	1/2	Experimental nitration/ distillation process	Explosion		Sudden oxidation (admission of air tc reduce vacuum without first cooling)
1091	Butadiene			Distillation column-leaky seal or pump	Fire		Fuel-air vapor ignited by open flame
1190	Ball Powder and Solvents		0/8	Distillation Process	Fire		No specification (puess-overheat and no agitation)

SUMMARY OF SELECTED ACCIDENTS WHICH OCCURRED IN A MILL

ASESB NO.	AGENT	AMOUNT (LB)	FATALITIES/ INJURIES	COMPONENT OR OPERATION	OUTPUT-TYPE	MAX. DISTANCE MISSILE/GLASS BREAKAGE (FT)	PROBABLE CAUSES
1130	Ballistite/50% Nitroglycerine	20 kg	1/2	Rolling Mill	Fire		Friction-foreign object
1073	M-5 Propellant; Cellulose water, Alcohol, wet Nitrocotton	130	0/2	Block Breaking Operation	Explosion		1) Pinching (broken belt entering process equipment)(fuel-air explosion possible) 2) Friction-blade impacting
391	Ammonium Perchlorate	1000	3/0	Crushing/Grinding Mill	Fire		1) Friction } foreign 2) Impact } particles
530	Potassium Perchlorate	125+125	1/0	Grinding Operation	Explosion-Fire	90/-	Hot spot from electric drive motor and contamination
606	Black powder	Tons	13/17	Granulation Section	Explosion		Friction
632	Igniter Pre-mix; Barium Peroxide, Zinc Stearate, Red Toner		1/7	Ball Mill	Explosion		Chemical decomposition/ unstable peroxide and moisture
1039	X-8 Propellant		0/0	Receiving Trough/ Rolling Operation	Fire		1) Friction 2) Contamination
685	Black Powder	1600	4/0	Wheel Mill	Explosion	400-750/5 miles	Unknown, no specification
940	Zerconium, Lead Dioxide, Binder, Igniter	300 g	0/2	Grinding/Mortar and Pestle	Flash Explosion		1) ESD } impact sensitivity>140 cm 2) Friction } temp of explosive >250°C
1607	Black Powder (rework, low nitrate fuze powder)		1/3	Milling Operation	Explosion		Friction scraping solidified explosive
1116	Phyto-pharmaceutical powder composition			Grinding/Filtering			1) Mechanical friction 2) ESD 3) Electrical spark
91	Rifle Powder	400	0/0	Wheel Mill Operation	Explosion		Friction-plough in contact with bed plate causing increase of heat

SUMMARY OF SELECTED ACCIDENTS WHICH OCCURRED IN A MILL (cont)

ASESB NO.	AGENT	AMOUNT (LB)	FATALITIES/ INJURIES	COMPONENT OR OPERATION	OUTPUT-TYPE	MAX. DISTANCE MISSILE/GLASS BREAKAGE (FT)	PROBABLE CAUSES
189	Black Powder	3000	0/0	Corning Mill Building	Explosion		1) ESD 2) Friction (bearing machine parts) 3) Foreign material in mix
202	Magnesium	100	0/0	Hammer Mill	Fire		Electrical-from lightning
548	Black Powder	500	0/0	Wheel Mill	Explosion		Unknown, no specification
771	Sodium Nitrate-Black Powder	805	1/0	Wheel Mill	Explosion		Unknown, no specification
867	Gun Powder	330	1/0	Granulating Roller Machine	Explosion		Unknown, no specification
649	Black Powder	500	0/0	Corning Mill	Explosion-Fire	1300/1 mile	Friction-foreign metal object between mill rolls ignited dust
781	Sodium Nitrate Black Powder	650	0/0	Wheel Mill	Explosion		Wheel Slippage-Friction
782	Black Fuse Powder	3700	0/0	Corning Mill	Explosion	350/1300 building demolished	Unknown
1277	M13	50	0/0	Stokes Granulator	Fire		1) Friction between agitator and screen 2) Abrasion nature of binding agent due to natural evaporation
1231	Ammonium Percholrate		0/0	Sneco Vibra-Energy Grinding Mill	Explosion		a) Adiabatic air compression b) Localized buildup near flanges fric tion c) Foreign particle
504	Green Charge Mix { Pot. Nit. Charcoal sulphur	312	0/0	Wheel Mill #2	Explosion-Fire	310/intra-plant	Unknown

SUMMARY OF SELECTED ACCIDENTS WHICH OCCURRED IN A MILL (concl)

ASESB NO.	AGENT	AMOUNT (LB)	FATALITIES/ INJURIES	COMPONENT OR OPERATION	OUTPUT-TYPE	MAX. DISTANCE MISSILE/GLASS BREAKAGE (FT)	PROBABLE CAUSES
1295	Ammonium Perchorlate		0/0	Raymond Grinder	Explosion	Bay area/ Building	Friction initiation due to contamination of bearing hub oil (hydrocarbon)
1316	N-S Paste Slurry			Expeller Mill	Explosion		Mechanical or chemical initiation(?) Friction
693	Sodium Nitrate Black Powder Composite			Wheel Mill	Explosion	223/-	Friction-too dry powder

SUMMARY OF SELECTED ACCIDENTS WHICH OCCURRED DURING PRESSING

ASESB NO.	AGENT	AMOUNT (LB)	FATALITIES/ INJURIES	COMPONENT OR OPERATION	OUTPUT-TYPE	MAX. DISTANCE MISSILE/GLASS BREAKAGE (FT)	PROBABLE CAUSES
110	¢83 Grenade MKII	--	-/-	Unloading mold	Fire		Friction (pinched explosive between two metal moving surfaces)
365	Explosive "D"	25	?	Press loading into shell	Explosion		Unknown
412	Smoke Composition PN507 (into No. 19 smoke container)		-/-	Pressing operation	Fire-Explosion		Friction (metal-metal contact)
449	Tetryl booster		0/3	Pelleting operation	Explosion		Unknown
484	Black powder	25		Pressing-15 in. horizontal	Explosion		Guess-friction
683	Black powder		1/0	Pressing operation		500/3-1/2 miles	Unknown-no specification
1341	Semi-gelatin dynamite		4/2	Cartridging machine	Explosion	300-400/-	Unknown-no specification
1379	Composition A5		0/0	Remote consolidation press	Explosion		Unknown-no specification
1381	40 mm Illuminating Cartridge		0/1	Consolidation press operation	Explosion		Unknown-no specification
1397(T)	55 gr PBSG-12		2/1	Pressing operation	Explosion		Friction
1407(T)	Composition A5		0/0	Consolidation press	Explosion		Unknown-no specification, guess-friction
1409(T)	Composition A5	81 gr	0/0	Penwalt press	Explosion		Unknown-no specification
1416(T)	Composition A5		0/0	Penwalt press	Explosion		Unknown-no specification
1421	PB-HMX	24 gr	0/0	Stokes pelleting press	Explosion		Friction
1597	Composition A-3	4.1	0/0	Denisson hydraulic press	Explosion		1) Friction (foreign article 2) Pinching cracking during press operation

SUMMARY OF SELECTED ACCIDENTS WHICH OCCURRED DURING PRESSING (cont)

ASESB NO	AGENT	AMOUNT (LB)	FATALITIES/ INJURIES	COMPONENT OR OPERATION	OUTPUT-TYIE	MAX. DISTANCE MISSILE/GLASS BREAKAGE (FT)	PROBABLE CAUSES
1600	RDX	12	0/1	Cherry barrel Rotary Press (pressing)	Explosion		Equipment failure, (guess friction)
1610	Lead-Azide, Lead Styphnate, Detonator manufacture		0/0	Final press house	Explosion		Unknown, no specification
1648	PEXN		0/0	Booster press cell operation	Explosion		Not clear
1663	Composition A5	10	0/4	Consolidation pressing, multistation rotary press; Colton model 270-18	Explosion		1) Friction, 2) Impact } mechanical equipment failure
1674	Igniter Mix	400 gr	0/2	Compacting/ pressing. Stokes Pelletizer Press	Explosion		Friction
1689	AFX 903 Experimental	53 gr	0/0	Pelletizing, 100 ton hydraulic Ram press	Explosion		Friction (foreign object inclusion)
99&100	Nitroglycerine gun cotton, ammonium nitrate and sodium nitrate		6/12	Press operation	Explosion		Friction (between cylinder wall and ram)
264	Tetryl booster charge		1/6	Pressing	Explosion		Friction (from ram)
320	Dynamite nitroglycerine		3/0	Cartriding/ pressing	Explosion		1) Friction from guide rods 2) Overheated from tester
1192	Flare composition	50	0/1	Pressing operation	Fire-Explosion		Unknown, no specification
1479	MK 48 Mod		0/0	Remote pressing operation	Explosion		1) Friction, 2) Impact } mechanical failure
705	Explosive-no specification		0/0	Rotary pelleting press	Explosion		Unknown, no specification

SUMMARY OF SELECTED ACCIDENTS WHICH OCCURRED DURING PRESSING (cont)

ASESB NO.	AGENT	AMOUNT (LB)	FATALITIES/ INJURIES	COMPONENT OR OPERATION	OUTPUT-TYPE	MAX. DISTANCE MISSILE/GLASS BREAKAGE (FT)	PROBABLE CAUSES
709	Alco Pellets for JATO Unit igniters (KCL_4 + A-)		0/1	Pelleting press	Explosion		Unknown, no specification
710	Black powder		2/2	Pressing operation	Explosion	500-600/1300	Unknown, no specification
722	8.6 grain AZY detonators, A Composition plus Lead Azide		0/1	Pellet extracting machine	Explosion		Friction (surplus material added)
752	Ammonia Gelatin 40%	4200	4/0	Gelatin cartridge house	Explosion	300/2 miles	Unknown, no specification
764	Charge increment for 40mm projectile			Pressing increment Dennison Consolidation press	Explosion		Friction
791	105mm shell (C-4)	7.25	0/0	Remote press-operation at 1527 psi	Explosion		Not determined
812	Rokrift: nitroglycerine and nitroglycerine	200	4/15	Miller Dann cartridging machines	Explosion		Friction (a) screw - container misalignment (b) box - explosive material interface Impact (a) mechanical failure (b) cartridge box dropped
814	Gelatin Dynamite	245		Gelatin cartridging house Starrett Gelatin machine	Explosion	550-650/800	Unknown, no specification
909	Gelatin		4/0	Cartridge machine house		300-750/3/4 mile	Unknown, no specification
918	Potassium Nitrate, Boron Laminac, Composition		0/2	Slugging/compacting	Explosion		Friction (pinching) Impact

SUMMARY OF SELECTED ACCIDENTS WHICH OCCURRED DURING PRESSING (cont)

ASESB NO	AGENT	AMOUNT (LB)	FATALITIES/ INJURIES	COMPONENT OR OPERATION	OUTPUT-TYPE	MAX. DISTANCE MISSILE/GLASS BREAKAGE (FT)	PROBABLE CAUSES
943	M17 Propellant	90	0/0	Faraguhar Vertical Blocking press (at 2000 psi) 11-3/4" diameter	Explosion		Adiabatic compression, (of vapors)
1016	Flare composition (igniter, fire clay, dry flare) Experimental		0/2	Pressing-Dennison Multipress	Fire		1) Friction/impact 2) Static spark
1063	PBX	2-1/2	0/0	Pelleting-Kur Lehner Single Action Press	Explosion		Impact/friction (mechanical failure of press)
1171	Alco Pellets for Tartar igniters		2/0	Pelleting press	Explosion		Friction (between turning table and wedged end plate)
386-1	Picric Acid			Pressing operation	Ignition		Impact - worker dropped base of mold into picric dust
386-2	Picric Acid			Pressing operation	Flash ignition		Impact
1279	NC		0/6	Dehydration press	Explosion		Friction-caused by misalignment of ram and NC block with liner
677	TNT	1/2	0/3	Plunger-die matrix	Explosion		Die-plunger misalignment causing frictional initiation
679	Tetryl pellet		-/-	KUX pelleting press	Explosion		Frication/impact; misalignment of ram; mechanical malfunction
687	Tetryl pellet		-/-	KUX pelleting press	Explosion		Impact: friction
711	TNT			Press	Explosion-Fire	125/-	Mechanical malfunction; metal-to-metal contact
754	Nitroguanadine green powder area (solvent vapor shot)		0/0	Pre-blocker press	Explosion		1) Autoignition of vapors 2) Impact

SUMMARY OF SELECTED ACCIDENTS WHICH OCCURRED DURING PRESSING (cont)

ASESB NO.	AGENT	AMOUNT (LB)	FATALITIES/ INJURIES	COMPONENT OR OPERATION	OUTPUT-TYPE	MAX. DISTANCE MISSILE/GLASS BREAKAGE (FT)	PROBABLE CAUSES
946	Flake TNT		0/0	300 ton transfer press	Explosion		Friction: 1) Extrusion-side of ram & die wall 2) Impact-ram & fractured die 3) Foreign material 4) Misalignment
1506	Composition A-3	Single Pellet	0/2	Stoke pelleting press	Explosion		1) Foreign material 2) Failure of punch 3) Excessive pressure due to excessive pellet buildup on-ram
1241	M-1 Propellant		0/1	Farguhar vertical blocking press	Fire-Explosion		Compression of entrapped air and solvent vapor causing autoignition (ethyl ether, ethyl alcohol)
802	M7 (solventless)	38		15" R.D wood	Explosion	141/-	--
718	Matador rework powder (rejected grains)	7 gr		Press	Explosion	20/-	Contamination of seal-line interface in die assy. Ram was stationary press-gate and die-assy as the focal point of detonation, compression of heel on 7 grain charge initiation possibility
653	C.E. perforated pellet	2 oz. pellet	0/0	Porter press	Explosion		Foreign particle inside press
377	TNT			Press	Explosion-Fire		Expl. dust and pulley slipping-friction initiation
757	Flake TNT	16 gr	0/0	Stokes pellet press	Explosion	Damage to immediate area	Foreign metal initiated (friction) during operation of press
10	Gunpowder	2 pellets + 40 lb (hopper)	0/2	Press	Explosion		Loose powder ignited by friction, pressure between extracting shoe and mold

SUMMARY OF SELFCTED ACCIDENTS WHICH OCCURRED DURING PRESSING (cont)

ASESB NO.	AGENT	AMOUNT (LB)	FATALITIES/ INJURIES	COMPONENT OR OPERATION	OUTPUT-TYPE	MAX DISTANCE MISSILE/GLASS BREAKAGE (FT)	PROBABLE CAUSES
943	M17 Prop Mix	90 available (top of block initiated)	0/0	11-3/4 vertical blocking press	Explosion		Ignition of solvent-vapor by adiabatic compression
1127	Black powder	(6) 30 mm pellets	0/1 burn	Press	Explosion-Burn		Unknown
975	Black powder	100 + 1500 charge house	0/0	Pellet press	Explosion-Fire	Immediate area	Unknown (possibly friction)
965	Boron - P. nitrate igniter comp	24 pellets (2 lb) + 7 lb on table	0/0	Stokes rotary press	Explosion-Fire	Immediate area	Friction between die and upper punch
699	Black powder			Block press	Explosion		Rapid compression of solvent vapors caused heat build-up to initiate explosive
612	Double base Nitrocotton-51.4% Nitroglycerine 42.9% (solventless)	70	0/0	15" R.D cordite press	Explosion-Fire	Limited to building 47/-	Compression of powder under flappers when pressed against room face
1048	Butadiene-M-Ammonium Nitrate	75	0/0	1070 ton extrusion press	Fire	Immediate area of press	Autoignition-adiabatic compression due to breakdown of limit switch controlling initial ram
1082	ABL 2056-D propellant		0/0	Watson-Stillman finishing press	Explosion	Immediate bay area	Ignition of solvent vapor vapors-adiabatic compression
833	ARP double base casting powder	"2 blocks"	1/0	Finishing press	Explosion	Immediate bay area	Adiabatic compression of solvent vapor (ether alcohol) due to blockage of vent
801	M-7			15" R.D. wood press	Explosion-Fire	625/	
1226	M-30 propellant			Farquhar 12"	Explosion-Fire	Immediate bay area	Adiabatic compression of entrapped vapor

SUMMARY OF SELECTED ACCIDENTS WHICH OCCURRED DURING PRESSING (concl)

ASESB NO.	AGENT	AMOUNT (LB)	FATALITIES/ INJURIES	COMPONENT OR OPERATION	OUTPUT-TYPE	MAX. DISTANCE MISSILE GLASS BREAKAGE (FT)	PROBABLE CAUSES
1212	Double-base casting powder	140/180	0/0	Finishing press	Explosion-Fire	Immediate area within building	At die and bottom of press basket surface) lateral motion-friction
1387	RDX pellets	1	1/3	Press	Explosion		1) Adiabatic compression of air trapped in wax or interface of wax and propellant-hot spot 2) Contaminated area-interface (propellant leakage) causing heat of friction initiation
1193	N4 (solvent-less) double base composition	100 (press) 707 (stored)		Ferquhar 15" horizontal press	Explosion	Immediate building	Friction initiation - due to foreign metal inclusion between ram and basket
1377	C-3 prop	60	0/0	Blocking press	Fire	Damage in immediate building	
64	SF62(4x.4 pellets)	165 gr	0/1 (burn)	(3) Triple punch Norsam presses	Explosion	Immediate area near punch	Perforating needle failue failure introduced as foreign article
714	Powder		2/5 (burn)	Horizontal finishing press	Flash Fire		During extrusion process
1013	AlClo	353 gr	0/1 (burn)	400 ton compaction press	Explosion-Fire	Minor damage	Misalignment of die mandrel & retaining mandrel causing interference (metal-metal)
862	Casting powder 0.1.C		0/0	Pawfoot press	Explosion (minor)	Superficial damage	Adiabatic compression - solvent vapor
1040	T36 prop	50 (rework)		Blocking press	Explosion		1) Heat by friction caused by interaction of ram head and cylinder wall and included powder buildup at inlet face (normal) 2) Friction at cylinder interface caused by mechanical failure at lower end of ram
1174	X-8		1/3	Lenspeed roll	Flash Fire		Unknown

SUMMARY OF SELECTED ACCIDENTS WHICH OCCURRED DURING FILLING

ASESB NO.	AGENT	AMOUNT (LBS)	FATALITIES/ INJURIES	COMPONENT OR OPERATION	OUTPUT-TYPE	MAX. DISTANCE MISSILE/GLASS BREAKAGE (FT.)	PROBABLE CAUSE
38	Detonators-Lead Styphnate	45-15 grain each	1/2	Transferring detonator to tray	Explosion-Fire	Immediate Area	1) Impact 2) ESD (unlikely 75% humidity)
58	TNT (Bomb)	150	0/2	Loading into wooden trays	Explosion		Impact
63	5 grain A/2 Detonators	15-5 grain each	0/1	Transfer Operation	Explosion		Impact
66	Igniters-S.R. 371 C	3-232 grains each	0/4	Filling Operation	Reaction		Friction
358	Grenades-#36		1/0	Inspection-Loading	Explosion	Immediate	Impact (Human Error)
359	15 in. Shells Mix	500	5/0	Screw Filling Hopper	Explosion		Friction (foreign object in hopper)
362	Amatol (50/50)	1/2	0/4	Transfer Operation after filling 75 mm H.E. gas shell	Explosion		1) Friction 2) ESD
364	Amatol (80/20)	75	3/1	Screw-Filling Operation for Extrusion	Explosion	300/-	Friction (foreign matter or solidified Amatol in hopper screw feed)
369	Amatol (60/40) 100°C		1/3	Filling Operation	Explosion		Impact (impact of brass tool on solidified composition)
387	Amatol (50/50)		2/4	Loading Shell Operation	Explosion		1) Friction (metal-to-metal contact with hammer) 2) Impact
432	Lead Azide - Comp. A		0/1	Transfer of "overfilled" detonator by operator	Explosion		1) ESD 2) Friction
439	Dynamite	2000	4/0	Loading Machine	Explosion	-/1 mile	ESD (ungrounded machine, low humidity)
561	Lead Azide-CE Detonators		0/1	Filling Operation (supply bowl detonated)	Explosion		ESD (brass filling ladle came into contact with brass scoop
600	Lead Azide - CE increment for 8.6 grain A.Z.Y. detonators	200 grains total	0/0	Filling Operation	Explosion		Friction (moving parts)

SUMMARY OF SELECTED ACCIDENTS WHICH OCCURRED DURING FILLING (cont)

ASESB NO.	AGENT	AMOUNT (LBS)	FATALITIES/ INJURIES	COMPONENT OR OPERATION	OUTPUT-TYPE	MAX. DISTANCE MISSILE/GLASS BREAKAGE (FT.)	PROBABLE CAUSE
608	5 grain A-Z detonators	20-5 grain each	0/2	Filling Machine (delivery chute)	Explosion		Friction (tapping-attempt at disruption of blockage)
621	M17 Detonater		0/1	Filling Operation	Explosion		Friction (dust ignition on work table/sleeve interface) Impact (insertion of aluminum foil into detonator)
636	Lead Azide and C.E. 6 grain detonators		0/0	Filling Operation Hopper Charge Explosion	Explosion		Friction (between guide plate and charge plate)
639	Dynamite	10,000	0/0	Pack-house	Fire		ESD (powder in paraffin dip pot ignited)
641	Lead Azide 6 grain detonators		0/0	Loading Operation	Explosion		?
654	Flash Bombs	25		Weigh-Fill Operation	Fire		Friction
659	?	1100 grains	0/1	Filling Operation	Explosion		Friction (attempt at cleaning blockage with pin on charge plate)
663	Lead Azide	5	2/0	Filling Operation	Explosion		1) Friction, 2) Impact, 3) ESD
1204	Illuminent Comp.		1/6	Charge Removal from Blender	Explosion-Fire		Unknown - No specification
1371(T)	M2- Relay/ Delay Elements		0/5	Syntron Feeder	Explosion		Unknown - No specification
1375(T)	Zirconium, Lead, Ethyl Acetate	2½	1/0	Filling-Hopper Operation	Explosion		Unknown - No specification
1389(T)	NOL 130 Primer Mix		0/0	Dumping into Blender	Explosion		Unknown - No specification
1590	Lead Azide, 3 flasks	20mg/flask	0/1	Transfer Operation	Explosion		Impact (dropped tray)
1598	Primer		0/0	Syntron Hopper Bowl-during Dumping Phase	Explosion		Unknown - No specification

SUMMARY OF SELECTED ACCIDENTS WHICH OCCURRED DURING FILLING (cont)

ASESB NO.	AGENT	AMOUNT (LBS)	FATALITIES/ INJURIES	COMPONENT OR OPERATION	OUTPUT-TYPE	MAX. DISTANCE MISSILE/GLASS BREAKAGE (FT.)	PROBABLE CAUSE
1599	NOL Primer Mix		0/1	Transfer Operation	Explosion		ESD ?
1612	Nitrogly-cerine and Gun Cotton Gelatinizing	250 kg	5/0	Loading Drais-Mixer (manual)	Explosion		Unknown - No specification
1619	Nitrogly-cerine-Azotic Cotton			Loading Gendu Mixer	Explosion-Fire	300/600	1) Friction (mechanical failure) 2) ESD (low humidity)
1640	Nitrocellu-lose Powder		40/11	Loading Room	Fire-Explosion		Unknown - No specification
1641	Mark 95 Detonators		0/1	Transfer Operation	Explosion		Unknown - No specification
1650	HMX	100 g	0/1	Filling Operation into Nitrator	Explosion		Unknown - No specification
1664	AIA Pyro-technic	150 g	0/1	Transfer Operation	Explosion		ESD (improper ground)
1702	Detonators M223 Grenade Mix		0/1	Automatic Transfer Operation	Explosion		Unknown
1703	PA-100 Primer Mix	120 g	1/0	Transfer Operation	Explosion		Unknown-No Specification
1403 (T)	NOL Primer Mix, Lead Styphnate Dextronated Lead Azide, Tetrazene, Barium Nitrate, Antimous Sulfide	1.75	0/1	Unloading/Handling after Blending Phase	Explosion		ESD?
57	Detonator facility	4-5	1/1	Transfer Operation	Explosion		ESD
84	Igniter, Tetryl		12/50	Loading Operation	Explosion		Faulty Fuse Assembly

SUMMARY OF SELECTED ACCIDENTS WHICH OCCURRED DURING FILLING (cont)

ASESB NO.	AGENT	AMOUNT (LBS)	FATALITIES/ INJURIES	COMPONENT OR OPERATION	OUTPUT-TYPE	MAX. DISTANCE MISSILE/GLASS BREAKAGE (FT.)	PROBABLE CAUSE
88	Aluminum Flare Composition (reworking material)			Transfer Operation	Explosion		Heat (hot spot ignition from IC engine nearby)
119	White Phosphoric Igniter		0/2	Transfer/Handling Operation	Explosion-Fire		Spontaneous Ignition (defective fuse)
120	Smokeless Powder	4000	1/0	Filling Operation on Feed Hopper	Fire		1) Impact - mechanical failure of hopper 2) Friction - between screen and hopper
132	Powder (Howitzer Shell)		3/2	Transport/Handling Process	Fire-Explosion	250/-	1) Friction 2) Static electricity 3) Foreign substance
	Firecracker Mix - (Potash Ground alum, Antimony)			Filling Operation	Fire-Explosion		Friction (contamination in between floor cracks)
178	Bombs		5/21	Packaging/Shipping	Explosion		Friction (between nose plug and bomb casing during handling)
261	Signal Lights-Dry Pellets		11/14	Transfer Operation	Explosion		Unknown - No Specification
262	Primer (fulminate)	595	3/6	Packing/Snipping	Explosion		1) Impact 2) Friction
312	M52 Incendiary Bombs- Black Powder-Primers	No. = 3000	0/1	Assembly/Loading Operation	Fire		1) Impact 2) Friction (human error)
314	Rocket Signal Star Pellets		1/0	Filling Operation	Fire		Impact (fall)
323	Primer Expl.	35 (Cal 30/50)	0/15	Filling/Loading Operation	Explosion		Impact (carelessness)
334	Black Powder		0/1	Supply Filling Operation for Pelleting Machine	Explosion		Friction

SUMMARY OF SELECTED ACCIDENTS WHICH OCCURRED DURING FILLING (cont)

ASESB NO.	AGENT	AGENT (LBS)	FATALITIES/ INJURIES	COMPONENT OR OPERATION	OUTPUT-TYPE	MAX. DISTANCE MISSILE/GLASS BREAKAGE (FT.)	PROBABLE CAUSE
451	Pyrotechnic Green Star Mixture		0/2	Mold Transfer Operation for Press	Explosion		Friction
596	Nitroglycerine - Dynamite	1200		Transfer/Loading Operation at Tally Mix House	Explosion	400-1850/500	Impact (mishap during delivery)
1347	NOL 130 Primer Mix	1.77	0/1	Post-Blending Process-Transfer Operation	Explosion		Unknown - No specification Guess - friction
722	8.6 grain AZY Detonator, Lead Azide, Comp. A		0/1	Pellet Extracting Machine	Explosion		Friction (surplus material)
737	Composition: Magnesium, Aluminum, Potassium Perchlorate			Discharging material into Receiving Buckets	Explosion-Fire		1) ESD 2) Heat (Magnesium water reaction) 3) Vibrator failure
746	A. Z. Detonator	7-5 grain detonators	0/1	Assembly/Loading	Explosion		1) Impact (from pit stick) 2) ESD
747	303 Cartridge Caps Mercury Fulminate, Potassium Chlorate, Antimony Sulfide, Sealed Powder, Sulfur		0/1	Refilling Hopper Supply	Explosion		Friction
756	30 cal. Primers		0/1	V&O Primer Insert Machine Filling/Pouring Primer into a cup	Explosion	15/10	ESD (non-conductive shoes)
758	Rocket Grain Mark 16 (Black Powder)		0/1	Extraction/Removal of Rocket Motor via Air Blowout Machine	Explosion		Friction (between faulty igniter and front closure device)
762	Rocket Grain Mark 16 (Black Powder)		0/0	Extraction of Igniter with Blowout Machine	Explosion		ESD
766	Lead Styphnate	3	1/0	Preparation for Filling	Explosion		1) ESD (non-conductive arch supports) 2) Impact

SUMMARY OF SELECTED ACCIDENTS WHICH OCCURRED DURING FILLING (cont)

ASESB NO.	AGENT	AMOUNT (LBS)	FATALITIES/ INJURIES	COMPONENT OR OPERATION	OUTPUT-TYPE	MAX. DISTANCE MISSILE/GLASS BREAKAGE (FT.)	PROBABLE CAUSE
824	Photoflash Composition for 762 mm Rockets		0/0	Remote Filling/Loading Operation from Hopper	Explosion		1) Impact (foreign body) 2) Friction (from gate valve assembly or foreign particle)
859	Al. Detonators		0/2	Assembly/Insertion	Explosion		1) Friction 2) Impact (between cover plate and detonators)
861	A.S.A. Composition: Lead Azide, Lead Styphnate	16		Dumping/Unloading from Mixing Process	Explosion-Fire		1) Friction (mixing bag) 2) Impact
865	Wet lead trinitroresorcinate	4.4	1/0	Weighing/Handling at Drying Station	Explosion	164/82	1) Impact (dried out material state) 2) Friction
869	Electric Detonators			Assembly/Transfer Operation	Explosion		ESD (low humidity)
881	Experimental Propellant	850		Unloading/Dumping of Barrel Tumbler	Fire		Impact (spillage)
904	Gelatin	3000	6/1	Gelatin Pack House	Explosion		Unknown - No specification
921	Nike Hercules Motor (Propellant)		0/2	Transfer/Carry Operation	Fire	200/-	Severe Impact
950	Electric Delay Detonators		1/0	Detonator Manufacturing/ Handling		0/30	Unknown - No Specification
1011	Giant Gel, 40% Dope, Sulfur		0/0	Feed Hopper/Weigh Station	Flash Ignition		1) Impact 2) Friction (hard 6061 Al. and steel interface)
1030	Primer Mix NOL - 130	3/4 ounce		Loading/Filling with Jones Loading Machine	Explosion		Friction (manual; between scoop and recepticle)
1051	M1 Smoke Pots (matchhead mix)		1/0	Post Blend Emptying Operation	Explosion		Unknown - No Specification
1075	Grenade - Pyrotechnics Agent (unclassified)		0/3	Transfer/Handling Operation for Seaming Machine	Fire-Explosion		1) Friction (between tray and cabinet) 2) Spontaneous Ignition 3) Friction (between pail and metal cabinet)

SUMMARY OF SELECTED ACCIDENTS WHICH OCCURRED DURING FILLING (cont)

ASESB NO.	AGENT	AMOUNT (LBS)	FATALITIES/ INJURIES	COMPONENT OR OPERATION	OUTPUT-TYPE	MAX. DISTANCE MISSILE/GLASS BREAKAGE (FT.)	PROBABLE CAUSE
1134	Pyrotechnic Mix, Potassium Chlorate, Sulfur, Sodium Bicarbonate		1/0	Unloading Operation of Read 84-17B Vertical Mixer	Explosion		Impact
1163	WP Filled Grenade		1/4	Assembly/Insertion	Explosion		Unknown - No specification
116B	High Energy NIBEX Propellant (Zirconium)	150	0/3	Dumping into Hopper	Fire-Explosion		1) Impact 2) ESD - dust accumulation in hopper
1214	M406, 40 mm Round, Composition B, Composition A5	32 gr.	1/15	Assembly/Handling	Explosion	32/-	Defective fuse caused premature ignition
1296	M1 Multi-perforated Single Base Propellant		2/4	Post-Dumping/Removal of Empty Plug Buggies from Area	Fire-Explosion		1) ESD 2) Crushing, Impact
1302	Primer Electric Model 52, Lead Styphnate, Graphite Potassium Chloride, Barium Nitrate		0/5	Filling Process on Conveyor Belt	Explosion		Friction (metal-metal)
1325	MK43 Mod Rocket Grain (35% NG, 46% NC) N-5 Rocket Paste	1870	1/3	Charging/Unloading Operation for Blender Barrel	Explosion-Fire		1) ESD 2) Friction (vibrator)
1547	C4			Hopper Dumping Operation	Ignition		Unknown - No specification
25	Nitroglycerine	7500		Loading/Storage Operation	Explosion		Impact
17	ROP Cordite			Incorporating House/ Packing	Explosion		Unknown - No specification
1273	Gilsonite, Sulfur Aluminum Fines		0/2	Charging/Loading Feed Hopper	Fire		Unknown
792	2½" Olgen Seismograph		2/0	Gelatin Pack House	Explosion		Unknown - No specification

SUMMARY OF SELECTED ACCIDENTS WHICH OCCURRED DURING FILLING (cont)

ASESB NO.	AGENT	AMOUNT (LBS)	FATALITIES/ INJURIES	COMPONENT OR OPERATION	OUTPUT-TYPE	MAX. DISTANCE MISSILE/GLASS BREAKAGE (FT.)	PROBABLE CAUSE
1121	Gelatin Dynamite	400 kg.	7/0	Transfer Operation in Mix House	Explosion		Impact
1126	Explosive Blasting Cap		0/50	Manufacturing Process	Explosion		Unknown - No specification
1141	Gelatin	3000		Gelatin Pack House	Explosion-Fire	1000/2000	Unknown - No specification
386-4	Picric Acid			Handling/Transfer Operation	Fire		Friction - friction between metal container and wall interface
1340	ANFO AN Prills	50M 5M		Howe Richardson Bagging Machine	Fire		Unknown - fire at diesel fuel area
349	Fire Gun Powder	2800	5/2+	Dryhouse (handling)	Explosion	350/	ESD - explosive dust explosion
631	Lead Azide	10 grain detector	0/1	Unloading moulds from extraction unit	Explosion	5-10'/--	Invested mold was brought in contact with surplus: explosion on top of the extraction machine
786	Ammonium Dichromate	150	0/0	Aluminum drum collector	Fire		1) Spark impingement of particle against metal valve on receiving can (ungrounded) 2) Heat of friction - V belt 3) Air lock valve blade became overheated due to friction
755	Multi-perforated single base M10 cannon powder and graphite dust	3000 and additional 2000	2/3	Loading preblender hopper	Explosion-Fire	900/4500	1) Static discharge due to powder impingement

SUMMARY OF SELECTED ACCIDENTS WHICH OCCURRED DURING FILLING (cont)

ASESB NO.	AGENT	AMOUNT (LBS)	FATALITIES/ INJURIES	COMPONENT OR OPERATION	OUTPUT-TYPE	MAX. DISTANCE MISSILE/GLASS BREAKAGE (FT.)	PROBABLE CAUSE
615	Composition A	--	0/0	Filling Unit	Explosion	--	Friction-metal-metal contact
892	Nitrogly-cerine, TNT, Nitrocotton, Dynamite, Dope	350/300/ 150/2700 /760	0/0	Dope Handling Unit	Fire-Explosion	400/1500	Unknown
569	Smokeless Powder	130,000	9/0	Filling Bin	Fire	--	1) Friction-metal-metal contact by opening slide gate on car 2) ESD
807	Black powder potassium nitrate	3500	2/0	Pack House	Explosion	100/-	--
1128	Powder dyna-mite	4620	2/4	Packing House	Explosion	900/-	Friction? container floor spark
1218	Lead Azide	15	1/13	Weighing Filling Operation	Explosion	Immediate Building	1) Impact initiation (dropping freon spray nozzle) 2) ESD 3) Friction initiation (Spatula and compound)
777	Photoflash Powder	16 and add 40		Loading Machine	Explosion	475/470	Impact or friction initiation
887	Hi-speed	1100	2/2	Cartridging Machine	Explosion	600/600	Unknown
1339	Nitrogly-cerine	7000	1/13	Loading Operation	Explosion	1200/3200	Unknown
1033	Dry nitro-starch	100	1/0	Unloading dryer	Explosion	75/0	Unknown
776	Photoflash Powder	25	0/3	Loading/filling machine	Explosion-Fire	Immediate Area	Static discharge
994	Petrogel #1		0/0	Filling/packing	Fire Flash	--	Sliding catch box over contaminated propellant
1272	M9 Propellant	--	2/32	Loading Machine	Fire-Explosion	--	Mechanical malfunction
1448	M9 Propellant		0/3	Loading perry accofil machine	Flash Fire	NA	Friction of exposed sensitive explosive

SUMMARY OF SELECTED ACCIDENTS WHICH OCCURRED DURING FILLING (concl)

ASESB NO.	AGENT	AMOUNT (LBS)	FATALITIES/ INJURIES	COMPONENT OR OPERATION	OUTPUT-TYPE	MAX. DISTANCE MISSILE/GLASS BREAKAGE (FT.)	PROBABLE CAUSE
3	Detonators	No. 13 detonator plug 190 grains	1/0	Breaking down detonators (dismantling)	Explosion	10/-	Striking detonator with a sharp tool and hammer
952	Ammonium perchlorate developmen-tal	--	3/1	emoving oxidizer lurry from tank	Explosion	--	Probing of packed sludge with a rod (friction/impact)

SUMMARY OF SELECTED ACCIDENTS WHICH OCCURRED DURING MIXING

ASESB NO.	AGENT	AMOUNT (LBS)	FATALITIES/ INJURIES	COMPONENT OR OPERATION	OUTPUT-TYPE	MAX. DISTANCE MISSILE/GLASS BREAKAGE (FT.)	PROBABLE CAUSE
390	Acetone Solvent and Paste		0/0	Melvin Incorporators	Fire		Friction (drive gear, clutch slippage and dust contamination)
499	Smokeless Powder (40 mm cannon)	154,450	3/13	Blending-Hopper Dumping Operation	Fire	1800/-	1) Impact } spark from 2) Friction } dropped article
531	Nitroglycerine, low Nitro, Amonium nitrate dynamite	75 1250	2/0	Mixing Operation-Talley Machine	Explosion	600-1100/ 1300	Unknown-No Specification
580	Primer Mix Lead sulphocyanate, Antimony sulfide, PETN	12	0/0	Frankford Arsenal Blending Machine	Explosion		Friction (a) due to overmixing of dried out composition (b) foreign article
592	Nitroglycerine			Mix House, Transfer Buggy Explosive	Explosion		Unknown - No Specification
627	Primer Mix #90, lead sulphocyanite, antimony sulfide, PETN, Potassium Chlorate	12	0/0	Blending Operation	Explosion		Friction
674	Smokeless Powder		2/0	Mixing Operation	Fire-Explosion	100/-	Unknown-No Specification
684	Mixed Dope		12/4	Mixing/Screening	Fire-Explosion	625-1100/ 800	Unknown-No Specification
694	Detonite	500	0/0	Mixing Operation	Explosion	150-300/ 4000	Unknown-No Specification (guess friction)
1352 (T)	Barium Chromate, Boron VAAR (90%, 9%)		0/0	Simpson Mixer	Fire		Unknown-No Specification
1365 (T)	NOL 130 Primer Mix	2½	0/1	Blending Operation	Explosion		Unknown-No Specification (guess friction)
1380 (T)	NOL 130 Primer Mix		0/0	Remote Blending Operation	Explosion		Unknown-No Specification

SUMMARY OF SELECTED ACCIDENTS WHICH OCCURRED DURING MIXING (cont)

ASESB NO.	AGENT	AMOUNT (LBS)	FATALITIES/ INJURIES	COMPONENT OR OPERATION	OUTPUT-TYPE	MAX. DISTANCE MISSILE/GLASS BREAKAGE (FT.)	PROBABLE CAUSE
1392 (T)	M63 Igniter Mix	5	0/0	Hobart Mixer	Explosion		Unknown-No Specification
1393 (T)	Trip Flare Mix-M49A1 Barium Chromate, Vinyl Acetate, Alcohol Resin Binder	25		Preparation/ Mixing	Fire		Friction
1403 (T)	NOL 130 Primer Mix, Lead Styphnate, Dextronated Lead Azide, Tetrazene, Barium Nitrate, Antimony Sulfide	1.75	0/1	Blending-Charge Removal	Explosion		ESD
1470	Whistle Composition: Oxides of Carbon, Sodium and Potassium		1/0	Mixing/Blending	Explosion		Unknown-No specification (guess ESD)
1571	Dynamite	3000	6/3	Mixing Process	Explosion		Unknown-No specification
1593	Monopropellant NOS366		0/0	Formulation/ Mixing	Explosion		Unknown-No specification
1595	Tracer Mix	50	0/0	Mix Operation	Explosion		Unknown-No specification
1615	Wood Powder Ammonium Nitrate		0/2	Mixing Process	Fire		Unknown-No specification
1629	Magnesium Teflon			Blending Operation	Deflagration Fire		Thermal (chemical instability - water and megnesium)
1637	PETN	10-20	1/0	Spin/Mix Operation	Explosion-Fire		Unknown-No specification
4	Propellant		8/25	Mixing Operation	Fire-Explosion		1) Friction (mechanical failure of mix paddle) 2) Impact (failure of paddle on foreign object)

SUMMARY OF SELECTED ACCIDENTS WHICH OCCURRED DURING MIXING (cont)

ASESB NO.	AGENT	AMOUNT (LBS)	FATALITIES/ INJURIES	COMPONENT OR OPERATION	OUTPUT-TYPE	MAX. DISTANCE MISSILE/GLASS BREAKAGE (FT.)	PROBABLE CAUSE
24	Black powder	1700 (Total) 50-100 (Barrel)	2/0	Sweetie Barrel Pre-Blending Operation	Fire		1) Impact 2) Friction 3) ESD
45	Powder	137/300		Powder Mixing Department	Explosion		Friction
205	Aluminum Composition Flare Mix (Grain Alcohol, Sodium Acetate Barium Nitrate, Castor Oil)	100	3/2	Blending Operation	Fire-Explosion		1) ESD (Dust ignition) 2) Friction (Foreign material) 3) Spark-electrical (motor wiring short)
238	Smokeless Powder -Cannon Powder	100,000		Blender Operation	Fire		Unknown-No Specification
291	Black Powder Reject and Additives		0/0	Mixing Operation	Explosion	1 mile/	1) Friction 2) Solvent Evaporation Overheat?
300	Barium Peroxide, Powder Magnesium Powder Aluminum	3400g. 400g. 200g.	1/1	Mixing Operation	Explosion-Fire		ESD (operation not grounded) and dust contamination
308	Strontium Nitrate, Magnesium Bee Wax, Shellac	14.25	1/0	Sigma Blade Mixing Machine	Explosion	225/-	1) Bearing Friction 2) Foreign Material - Friction
767	Blasting Powder	200	0/0	Powder Mixing House	Explosion	-/600	Unknown-No specification
809	HMX Base	122	0/0	Baker Perkins Mixer		300/600	Unknown-No specification
837	Dynamite Nitroglycerine	1600 460	2/3	Dynamite Tally Mix Operation	Explosion	600/-	Unknown-No specification
871	Rocket Propellant and Solvent		0/0	Baker Perkins Mixer/Cleaning Operation	Fire		Friction (ignition of solvent vapors during scraping)
894	Rocket Propellant (solid) XM10	2000	0/0	Baker Perkins Sigma Blade Mixer	Explosion		Friction (blade-container and dust ignition from spark)

SUMMARY OF SELECTED ACCIDENTS WHICH OCCURRED DURING MIXING (cont)

ASESB NO.	AGENT	AMOUNT (LBS)	FATALITIES/ INJURIES	COMPONENT OR OPERATION	OUTPUT-TYPE	MAX. DISTANCE MISSILE/GLASS BREAKAGE (FT.)	PROBABLE CAUSE
902	Pyrotechnic Mixture: Sodium Nitrate, Magnesium Laminac Binder, Acetone, Air Vapors	3½	0/0	Mix Operation-Simpson Mixer	Explosion		1) Friction (foreign material within container 2) Hot Spot Ignition (hot motor and dust ignition)
905	Gelatin	4000	3/2	Mix House	Explosion	800/1-1½ miles	Unknown-No specification
951	Rocket Propellant Composite NG, Polyester, Alum Powder, Ammonium Perchlorate	10		Mixing Operation Baker Perkins Sigma Blade	Explosion		1) Friction (between blade and food or foreign object) 2) Thermochemical Exothermic reaction-Instability
977	Rocket Propellant Composition (Type HDDR-A) Alum Powder, HMX Slurry, Ammonium Perchlorate, Acetone, Alcohol		0/0	Readco Single Arm Double Blade Mixer	Fire		Friction (blade and bowl or foreign object)
992	Pyrotechnic Mixture, 7-27% Laminac, 23.9% Boron, 68.7% Potassium Nitrate, 500 grains of Trichlorate Ethylene		0/1	Mix Operation	Fire		1) Friction (metal-metal) 2) Friction (between solid contaminant on blade and metal bowl)
1001	Propellant Slurry Type MR382	350	0/1	Vertical Mixer with Turbine Blades	Explosion		1) Impact (foreign object) 2) Friction 3) Friction (seal-shaft contamination) 4) Cavitation
1003	Polysulphide Perchlorate Solid Rocket Propellant	2882	0/11	Baker Perkins Sigma Blade Mixer	Explosion		1) Frictional Heat (fuel oxidizer in packing gland range) 2) Friction (blade-wall) 3) Pinching(metal separation shift-packing interface, cracks, crevices)

SUMMARY OF SELECTED ACCIDENTS WHICH OCCURRED DURING MIXING (cont)

ASESB NO.	AGENT	AMOUNT (LBS)	FATALITIES/ INJURIES	COMPONENT OR OPERATION	OUTPUT-TYPE	MAX.DISTANCE MISSILE/GLASS BREAKAGE (FT.)	PROBABLE CAUSE
1021	Experimental Igniter Mixture		0/0	Blending-Twin Shell V Blender	Deflagration		1) Friction 2) Impact
1024	Tracer Composition XM313	50	0/0	Mixing Operation			1) Friction 2) Impact
1035	Composite Propellant Fuel and Powdered aluminum oxidizer mixed of perchlorate			Mixing Process	Fire		1) Friction Overheat 2) Friction between discharge spout and line bin 3) Friction from bin vibrator
1064	Propellant, ammonium perchlorate rubber composition			Mixing Operation Read Horizontal Mixer	Explosion		1) Impact 2) Friction
1067	Tracer Mix XM13		0/0	Simpson Mixer	Explosion		1) Friction 2) Impact
1072	Igniter Mix Explosive	1000-1500	3/8	Mix House	Explosion		Unknown-No Specification
1096	Lead Azide Calcium Stearate Jelly Mix			Mixing Cycle	Explosion		Unknown-No Specification
1114	Charcoal Mixers			Colloid Mixing Equipment	Explosion		Unknown-No Specification
1154	Phosphorous Ferrosilicon and Flare related Alloys		0/1	Stirring/Mixing	Violent Reaction		Thermal-Runaway chemical reaction
1167	R&D Propellant	10	2/0	Horizontal Mixer-Sigma Blades	Explosion		Thermal-Runaway Chemical Reaction
1200	Propellant Mix (Miniature)		0/0	Batch Mixer	Fire-Explosion		Friction-blade-lining-foreign object
1204	Illuminant Composition		1/6	Blender-Charge Removal	Explosion-Fire		Unknown-No Specification
1237	R&D Solid Composition		0/0	Simpson Mixer	Fire		Friction-internal friction of Composition
1257	Hi-energy Propellant Hydrazine Di-Perchlorate	52	0/0	Baker Perkins Vertical Mixer			Friction

SUMMARY OF SELECTED ACCIDENTS WHICH OCCURRED DURING MIXING (cont)

ASESB NO.	AGENT	AMOUNT (LBS)	FATALITIES/ INJURIES	COMPONENT OR OPERATION	OUTPUT-TYPE	MAX. DISTANCE MISSILE/GLASS BREAKAGE (FT.)	PROBABLE CAUSE
1270	Gelatin Dynamite and Nitro-glycerine			Mix Operation	Explosion	350-800/1200	Unknown-No specification
1286	N5 Propellant Paste	880	0/2	Blender Dust Collector Roto-Clone Apparatus	Explosion		1) Friction 2) Pinching
1322	Slurry Mix		0/1	Hobart Mixer			Friction (metal contact between liner and paddle)
1206	Double Base Propellant (NC, amm. Perchlorate and Aluminum)	720	0/0	Mixer	Fire		Foreign objects (metal) frictional hearing of blade-object-lining interface
1261	TP-H1085 Propellant	4400	3/2 (fire)	Baker-Perkins mixer 300 gal (scrape down)	Fire		Ignition of ammonium perchlorate/sublimed recrystallized ferrocene by heat of friction. Action: steel spatula/liner during scrape down
1289	Single base multiperforated powder	5000		Mixer (discharge into buggy)	Explosion		Ignition of dust granules by ESD
811	Propellant composite	500	2/0	Mixer	Explosion-Fire	(900/2000)	Foreign article-scraping tool inside mixer caused frictional heat initiation between blade and liner
964	Polysulfide perchlorate			Extruder	Explosion		Friction: 1) foreign object 2) blade and linear contact 3) solid buildup
657	Propellant Carbon black Nitrocellulose Ammonium perchlorate		0/0	Mixer	Explosion	400/-	Too low a content of solvent in mixture (dry friction)

SUMMARY OF SELECTED ACCIDENTS WHICH OCCURRED DURING MIXING (concl)

ASESB NO.	AGENT	AMOUNT (LBS)	FATALITIES/ INJURIES	COMPONENT OR OPERATION	OUTPUT-TYPE	MAX. DISTANCE MISSIL./GLASS BREAKAGE (FT.)	PROBABLE CAUSE
1473	Casting (20% scrap powder)	620	0/0	Talley mixer	Fire	-/-	Friction between blade and lining
920	Dynamite-Nitroglycerine	1900 700	2/5 (frag)	Talley machine	Explosion	600 ft/3 mi	Unknown
1309	C-3 casting powder		0/0	Mixer	Fire-Explosion	Bldg only	Foreign particle/failure or blade; friction/impact
896	Composite propellant	98 lbs	0/2 (flash burn)	20 gal Baker Perkins mixer	Explosion	-/-	Foreign article between blades and liner caused frictional heating/impact
878	Polysulfide base TRX 110C propellant		-/-	200 gal Baker Perkins mixer	Explosion-Fire	150 ft/-	1) Blade clearance .088/.109 in - friction 2) Static spark (gas leakage observed-ammonia perchlorate and fuel)
729	Composite (nitroguanidine, K_2SO_4)	200 3.9	0/2 (fire)	Mixer charging	Fire		Sigmablade-frictional heating
1310	NACO propellant single base	"8 blocks"	2/1 (burn)	Mixer	Fire		1) Foreign material-friction 2) Dehydration of mixture increasing sensitivity for impact 3) Metal-to-metal blade lining contact caused by initial deflection of resistive force with NACO blocks
472	M7 propellant	450 lb	1/1 (blast) victim thrown 120 yards	Mixer	Explosion	-/-	Friction-blade-lining pressing of dry potassium perchlorate pre-blend (.025 in. clearance)
1080	Polybutadiene amm. perchlorate and MAPO (solid propellant)	280	0/0	Mixer	Fire		Spontaneous autoignition of MAPO - no blades in mixer

SUMMARY OF SELECTED ACCIDENTS WHICH OCCURRED DURING MAINTENANCE

ASESB NO.	AGENT	AMOUNT (LB)	FATALITIES/ INJURIES	COMPONENT OR OPERATION	OUTPUT-TYPE	MAX. DISTANCE MISSILE/GLASS BREAKAGE (FT.)	PROBABLE CAUSES
355a	Fuel-Vapor (Alcohol)	5 gal. can	0/3	Alcohol Tank	Explosion		
355b	Tetryl Lead			Cleaning Operation on Lead Crystallizer	Fire-Explosion		Direct Flame on Trapped Explosive
355c	Tetryl			Discarding Lead Pipe	Explosion		Impact - Lead Pipe and Tetryl
355d	Tetryl			Maintenance Operation on Valve Seat	Explosion		Direct Flame Exposure
355h	Tetryl		0/3	Maintenance/ Soldering on Contaminated Tetryl Car	Explosion		Thermal - Heat Application
355e	Tetryl			Maintenance/ Replacement of Lead Cover	Explosion		Unknown
355g	Mixture-M3 Flare			Welding	Explosion		Hot Spot - Contaminated metal mold
363	Boosters - MK III, IIIA (TNT and Tetryl)		0/0	Soldering	Explosion		Thermal - overheated and dust available
589	Lead Azide mixture		1/1	Spray Painting Operation of 120 MM Filled	Explosion		1) ESD 2) Friction (foreign particle)
1370 (T) and 1328	NOL 130 Primer Mix		0/1	Maintenance Operation on Jones Loading Machine	Explosion		Friction
1383(T)	Lead Azide			Maintenance Operation in Storage Area	Explosion		1) Impact 2) ESD
1414(T)	TNT dust		0/0	Cleaning/ Maintenance	Explosion		1) Friction 2) Mechanicsl Failure of Blower - Impact
1420(T)	Igniter Composition Mix		1/2	Maintenance Operation on Mixer	Explosion		Friction - contamination

SUMMARY OF SELECTED ACCIDENTS WHICH OCCURRED DURING MAINTENANCE (cont)

ASESB NO.	AGENT	AMOUNT (LB)	FATALITIES/ INJURIES	COMPONENT OR OPERATION	OUTPUT-TYPE	MAX. DISTANCE MISSILE/GLASS BREAKAGE (FT.)	PROBABLE CAUSES
213	Black Powder			Disposed Operation	Explosion		Unknown-No specification
244	Gun Powder		4/7	Salvage	Explosion		Unknown-No specification
266	Not Specified		0/5	Maintenance Operation for Press	Explosion		Unknown-No specification
315	Black Powder	~5000	5/2	Press Maintenance Operation	Fire-Explosion		Friction (metal-metal)
772	Experimental Explosive for 105 mm round		1/1	Maintenance/Cleaning	Explosion		1) Friction 2) Impact
871	Rocket Propellant and Solvent		0/0	Cleaning Operation/ for mixer	Fire		Friction-foreign material scraping and vapor ignition
931	Hydrogen Peroxide			Drainage/Disposal	Explosion		Flammable Fuel Ignition
973	Igniter Composition	1/4	0/1	Handling/Cleaning of Pelleting Press (Kur Lehnar)	Flash Ignition		1) Friction-between shoe and table 2) Pinching (compressing)
1025	Blasting Agent(fuel oil, ammonium nitrate)		0/26	Acetylene Torch Cut on Chute	Fire-Explosion	3 mile/5 mile	Open Flame
1062	M49 and M28B2 Artillery Primer		1/0	Maintenance Operation for Jammed Chute Component on Disposal Furnace	Explosion		1) Thermal 2) Impact (by operator)
1103	Aluminum Powder, Magnesium Perchlorate		0/2	Cleaning/Vacuuming	Deflagration	50/-	Friction and Dust Ignition
1112	Explosive Mixture Trialene (70% TNT 15% Hexogene, 15% Alum)			Maintenance	Explosion		Friction
1182	Chemical Filter Solution Water Methanol (50-50) and 5% Caustic		0/0	Decontamination/ Cleaning	Explosion-Fire		Fuel Air Vapor Ignition 1) Heat of Friction 2) ESD 3) Heat of Reaction 4) Friction-Sump Drain Actuation

SUMMARY OF SELECTED ACCIDENTS WHICH OCCURRED DURING MAINTENANCE (cont)

ASESB NO.	AGENT	AMOUNT (LB)	FATALITIES/ INJURIES	COMPONENT OR OPERATION	OUTPUT-TYPE	MAX. DISTANCE MISSILE/GLASS BREAKAGE (FT.)	PROBABLE CAUSES
1299	Bomb Fuses		1/D	Inspection/Boat Disposal Cleaning/ Maintenance	Explosion		Unknown-No specification
386-5	Picric Acid			Cleaning Operation	Fire		1) Impact 2) Friction
386-1D	Picric Acid Dust			Maintenance	Fire		1) Impact 2) Friction
386-13	Picric Acid			Cleaning/Maintenance	Explosion-Fire		Friction from tools
386-23	Picric Acid			Maintenance/Disposal	Explosion		Direct Flame - contaminated lead melt down
386-2B	Picric Acid			Maintenance/Melt down of metal in furnace	Explosion		Direct flame-scrap metal contaminated with picric acid
611	"Explosive"	3/4	1/5	Maintenance-conveyor system	Explosion	Immediate area	Cutting torch-localized heat on contaminated vacuum pipe
626	"Dry" Nitrocellulose		1/2	Repair and maintenance	Explosion		Cigarette/match ignition of contaminated underground pipe
41D	Contaminated waste water		1/0	Maintenance	Fire-Explosion		Waste water residue contaminated in basin. Fire initiated by friction from metal friction subsequent explosion of pipe caused by heating due to fire
356	Residual dry Nitrocellulose	5-10	0/5	Cleaning	Explosion		Swab initiated Nitrocellulose within pipe by friction
1234	Composition A-5	17-25 lbs + 25 + 5		Cleaning-maintenance	Fire-Explosion		Caused by striking vacuum kettle against garbage can to provide ignition by 1) Impact 2) Friction 3) ESD

SUMMARY OF SELECTED ACCIDENTS WHICH OCCURRED DURING MAINTENANCE (concl)

ASESB NO.	AGENT	AMOUNT (LB)	FATALITIES/ INJURIES	COMPONENT OR OPERATION	OUTPUT-TYPE	MAX. DISTANCE MISSILE/GLASS BREAKAGE (FT.)	PROBABLE CAUSES
1180	Double base triple base propellant		1/0	Maintenance-welding of bride block	Explosion		Welding of contaminated bride block sufficient to allow localized heat initiation
1227	Lead azide resudial crystals		1/0	Cleaning	Explosion		Impingement of contaminated residual sludge by hose stream caused foreign objects within to abrade LA crystals
789	Nitrocellulose colloid		1/.	Hardening still (cleaning scraping)	Flash Fire		1) Impact from brass scraper 2) Friction-employee standing on residue material 3) Decomposition of remains within still

SUMMARY OF SELECTED ACCIDENTS WHICH OCCURRED DURING MACHINING

ASESB NO.	AGENT	AMOUNT (LB)	FATALITIES/ INJURIES	COMPONENT OR OPERATION	OUTPUT-TYPE	MAX. DISTANCE MISSILE/GLASS BREAKAGE (FT)	PROBABLE CAUSES
1490	Nitrocellulose slurry	contaminated pipe section	0/1	Hacksaw blade	Explosion	10/-	Friction due to metal-metal contact
1499	Black powder (block)	41	0/1	Bandsaw	Fire		Friction between metal and black powder
1205	Propellant grain		0/0	Saw	Fire		Friction
1108	Reinforced grain (Minuteman rocket motor)	10	0/0	Vertical radial saw (hydraulic)	Fire		Inherent friction in machining of reinforced grain
735	Rocket grain		0/0	Dowel rod machine	Explosion	Immediate bay area	Friction initiation during machining
1254	Sparrow MX 38 mod 0 solid grain prop	91	0/1	1/32 Drill into prop	Fire		Friction heat buildup
959	Composition prop experimental Emerson-Cummings Epoxy resin catalyst alum. powder, pot. perchlorate	(cast) 3/4	(cuts) 0/1	Machining lathe	Explosion		Decomposition of Unknown experimental chemical
686	Mark 16 solid prop		0/1	Rotary saw	Fire		Spark initiation and (friction) heat buildup
899	Benite powder	41 strands	0/2	Bandsaw	Fire		1) Ignition of vapors due to friction caused by: a. Excessive saw speed b. Insufficient coolant flow c. Adherence of powder to saw and revolving under wheel of saw 2) Static spark ignition of alcohol vapors
602A	JON extruded MX22 grains	65	0/0	Saw	Fire-Detonation		Friction originated, drip vacuum enhanced spread of fire

SUMMARY OF SELECTED ACCIDENTS WHICH OCCURRED DURING MACHINING (concl)

ASESB NO.	AGENT	AMOUNT (LB)	FATALITIES/ INJURIES	COMPONENT OR OPERATION	OUTPUT-TYPE	MAX. DISTANCE MISSILE/GLASS BREAKAGE (FT.)	PROBABLE CAUSES
355f	TNT			Machining stirrers	Explosion		Friction
355h	Smokeless powder		0/0	Machining ram press	Explosion		Friction
692	AN150A2		0/0	Thermate drilling machine	Explosion		Friction & primer detonator initiation (detonator not secured prior to drilling)
1359	Comp B 60/40	4.5	0/0	Drilling operation	Explosion		Frictional heat buildup between workpiece and tool
1494	H.E. explosive	6		Drilling operation	Explosion		Friction between drill blade and fuze wall thrcads
1690	PFX	75	3/0	Machining rough billet 20" Monarch tracer lathe	Explosion		1) Friction 2) Impact
334(2d)	Primers		0/0	Drilling operation	Fire		1) Friction 2) Sparks
1037	Ammonium Perchlocate solid propellant	92	0/0	Cutting operation	Fire-Explosion		Frictional heat
633	Rocket propellant powder	56(initial) 548(total)	1/0	Cutting machine	Explosion		1) Friction-steel blade on machine ignited nitrogylcerine fumes or powder dust 2) Impact knife on machine guide causing shock initiation of nitro condensate
655	Mercury fulminate		1/2	Rebowling operation	Explosion		Inadvertent impact
1038	TPH 8126 composite propellant	total 7	1/2	Bandsaw	Explosion-Fire	12 ft/-	Ignition of propellant fines in lower guide blocks of bandsaw friction

SUMMARY OF SELECTED ACCIDENTS WHICH OCCURRED DURING EXTRUSION

ASESB NO.	AGENT	AMOUNT (LB)	FATALITIES/ INJURIES	COMPONENT OR OPERATION	OUTPUT-TYPE	MAX. DISTANCE MISSILE/GLASS BREAKAGE (FT.)	PROBABLE CAUSES
583	Experimental propellant			Pressing-extrusion combined chamber machine	Fire-Explosion		Unknown, no specification
1185	ASROC (Cruciform grain - rework)		0/0	Press-extrusion operation	Explosion-Fire	660/-	1) Powder contamination 2) Friction (powder-powder) 3) Heat of impact 4) Trapped air compression within press bucket
1385	M30-multi-perforated triple base propellant	30	0/0	Extrusion press	Explosion	Damage in press bay	
1376	Multibase casting powder	35/80	0/0	Extrusion press	Fire		
835	Double base solventless rocket propellant Navy X-8		0/0	Extrusion press	Explosion		
773	N5		0/0	Extrusion press	Fire-Explosion	Bay area	Heat generated by fire near press
913	Ammonium perchlorate			Lombard horizontal extruder	Explosion	2 cells completely demolished	1) Friction of moving parts 2) Adiabatic compression of trapped air 3) Heat rise during normal operation 4) Oxidizer entrapped between moving parts
937	N-5 formula (solventless double base slurry)		0/0	Expeller/extruder	Explosion-Fire	~860 ft/--	1) Friction generating heat hear torpedo head 2) Foreign material causing friction, pinching or impact

SUMMARY OF SELECTED ACCIDENTS WHICH OCCURRED DURING EXTRUSION (concl)

ASESB NO.	AGENT	AMOUNT (LB)	FATALITIES/ INJURIES	COMPONENT OR OPERATION	OUTPUT-TYPE	MAX. DISTANCE MISSILE/GLASS BREAKAGE (FT.)	PROBABLE CAUSES
1419	Triple base propellant	10	0/0	Extrusion press	Explosion	Damage to press, building	Foreign metal inclusion
976	Double base solventless	50	0/0	Watson-Stillman 15" horizontal extrusion press	Explosion-Fire	80/-	Friction; failure of Teflon ring seal; metal to metal contact; ram & press basket
690	M-10		0/2	Extrusion press	Explosion	Immediate area	1) Adiabatic compression 2) Rapid extrusion-localized overheating

SUMMARY OF SELECTED ACCIDENTS WHICH OCCURRED DURING SCREENING

ASESB NO.	AGENT	AMOUNT (LB)	FATALITIES/ INJURIES	COMPONENT OR OPERATION	OUTPUT-TYPE	MAX. DISTANCE MISSILE/GLASS BREAKAGE	PROBABLE CAUSES
671	Dope		0/1	Dope Mixer Screen	Ignition		Unknown - No Specification
1674	Lead Azide	1	0/0	Screening Operation	Explosion		1) Friction 2) Impact 3) ESD
92	Black Powder	6000		Screening/ Packing	Explosion-Fire		1) Friction (metal-metal) 2) Impact 3) ESD (not likely)
120	Smokeless Powder	4000	1/0	Screening House/Filling Hopper	Fire-Low Order Detonation		1) Friction (screen-hopper)
164	Black Powder	5000	2/5	Screening Operation	Explosion		1) Friction \ Mechanical Failure 2) Impact / 3) Spark Ignition
1115	Lead Azide	140 g.		Sifting-Sieve Operation	Explosion		Unknown - No Specification
1603	Lead 2:4 Dinitro Resorcinate	1.2	0/0	Sieving	Explosion		Unknown - No Specification
1659	Lead Azide	1	0/0	Screening Operation	Explosion		Impact
684	Mixed Dope	3000	12/4	Screening/Mix Operation	Fire-Explosion		Unknown-No Specification
721	Mercury fulminate	9 oz.	0/1	Sieving	Explosion		Impact/friction initiated striking of funnel with brush handle
785	Lead styphnate	1-1/2 lbs.	0/0	Jelly bag screener	Explosion	50/-	1) Impingement-during pouring of L.S. over screener 2) ESD

SUMMARY OF SELECTED ACCIDENTS WHICH OCCURRED DURING SCREENING (Concluded)

ASESB NO.	AGENT	AMOUNT (LB)	FATALITIES/ INJURIES	COMPONENT OR OPERATION	OUTPUT-TYPE	MAX. DISTANCE MISSILE/GLASS BREAKAGE	PROBABLE CAUSES
744	None-dynamite dope ingredients	--	0/0	Screener	Fire		Friction caused by foreign material in screen
751	None-dynamite dope ingredients	--	0/0	Screener	Fire		Electrical wiring
581	T9 powder pot. nitrate, ammonium picrate, aceton, ethylcellulose, zinc stearate, tri-calcium phosphate	1700 lbs. total in area	2/0	Blender/ screener unit and filling drums	Explosion		1) Frictional-metal-metal contact 2) Static discharge-all during operation
1184	Green smoke powder	--	--	Sifting	Explosion		Friction

SUMMARY OF SELECTED ACCIDENTS WHICH OCCURRED DURING STORAGE

ASES NO.	AGENT	AMOUNT (LB)	FATALITIES/ INJURIES	COMPONENT OR OPERATION	OUTPUT-TYPE	MAX. DISTANCE MISSILE/GLASS BREAKAGE	PROBABLE CAUSES
1338	Ammonium Nitrate		0/8	Storage Facility	Explosion		Unknown - No Specification
1378(T)	Pyroteehnic material		0/0	Storage	Fire-Explosion		Unknown - Lightning, heavy rains
97	DuPont Nitro-glycerine	3000	2/8	DuPont Frame Shed	Explosion		Unknown - No specification
224	Ammonium Sulphate-Nitrate	4500 ton		Storage	Explosion		Stability unknown
207	Ammonium Nitrate			Storage	Explosion		Unknown
313	AN-M40 Signal flares and smoke pots			Storage	Fire		Unknown
386-3	Picric Acid			Storage magazine	Fire		Friction
386-11	Picric Acid			Magazine	Fire		Friction
945	Nitro-glycerire	3000	0/0	Storehouse	Explosion	1000/plant area	Initiation by range fire
1079	Nitro-glycerine	7540	1/1	Storage Tanks	Explosion	1000/1 mile	Unknown ,friction possibility) deceased entered area with pail
528	Gun powder	36 tons	15/25	Storage	Fire-Explosion	1/4 mile 1-3/4 mile	Fire initiated
597	Powder	--	--	Storage	Explosion	--	Cigarette initiated
661	Nitrates a) Soda b) Ammonia	--	12/-	Storage	Fire-Explosion	Crater 22 ft Denth 6 ft	Unknown

SUMMARY OF SELECTED ACCIDENTS WHICH OCCURRED DURING DRYING

ASESB NO.	AGENT	AMOUNT (LB)	FATALITIES/ INJURIES	COMPONENT OR OPERATION	OUTPUT-TYPE	MAX. DISTANCE MISSILE/GLASS BREAKAGE (FT.)	PROBABLE CAUSES
280	Lead Azide-Tetryl for 6 gr detonators	12	1/0	Drying Operation	Explosion	20/-	ESD?
384	Picric Acid	1450(P)	0/22	Drying House	Fire-Explosion	750/2-5 miles	1) Friction (man's clogs walking through room) 2) Impact (.. of picrate of iron by man's shoes) 3) Overheated (steam pipe)
385(1)	Ammonium Nitrate (AN)			Crystallizing Pan Shed-Drying Process	Fire		Heat (contaminated bagging placed on steam pipe)
385(2)	AN			Evaporating Plant	Ignition		Heat (thermal ignition of contaminated asbestos covering steam pipe)
385(4)	AN			Drying Process	Reaction		Excess heat-steam pipe cover contaminated with AN
385(5)	AN			Drying Process	Reaction		Excess heat application-hot bricks on AN thin layer
385(6)	AN			Drying	Ignition		Heat (contamination)
385(7)	AN			Drying Operation	Explosion		Heat (steam pipe and contamination)
1346	Nitroglycerine	300	0/0	Drying Process	Explosion		Heat (high temperature over extended period, decomposition of nitroglycerine)
1468	Igniter Composition for M49A1 flare	2000	0/0	Drying/Storage	Fire-Explosion		Unknown-no specification
1560	Solventless sheet propellant		0/0	Preheating Operation	Explosion		Unknown-no specification
1570	Primer Ignition Assy M63; M59			Drying	Explosion		Unknown-no specification
1604	Shotgun Powder	250	0/1	Drying Operation	Fire		Impact (on layer of powder)
1613	118 Electric Detonators			Drying	Explosion		Overheat due to faulty thermostat

SUMMARY OF SELECTED ACCIDENTS WHICH OCCURRED DURING DRYING (cont)

ASES8 NO.	AGENT	AMOUNT (LB)	FATALITIES/ INJURIES	COMPONENT OR OPERATION	OUTPUT-TYPE	MAX. DISTANCE MISSILE/GLASS BREAKAGE (FT.)	PROBABLE CAUSES
1647	Red Water Facility-Thick Liquor		0/0	Drying/Heating	Explosion		Thermal (entrapped explosive material overheated)
51	DuPont Shotgun Powder	2080	2/0	Drier Operation	Fire		Friction
163	Black poweder	17,000	5/9	Dry House	Explosion		Unknown
218	DNT-TNT	25,000	36/2 (34 missing)	Drying Process	Explosion	2640/-	Heat (overheating)
270	Magnesium			Drying Operation	Fire		Heat (excess heat applied)
325	Ammonium Nitrate (1% H_2O)	4800	4/17	Evaporator Pan/ Drying Process	Explosion		Thermal (ignition from overheated lub oil in air agitator die)
1482	Propellant M26	7	0/0	Hot Pack Oven/ Drying Operation	Fire		Thermal (thermostat malfunction causing overheat)
728	M80 Firecracker Composition; Potassium Perchlorate, Alum flakes, Sulphur Antimony Sulphide		11/50	Drying Oven	Explosion-Fire	-/1600	No specification, guess-overheating
893	6 Nike and 10 Honest John Motors (propellant)		0/0	Curing/Drying Building	Fire		Electrical (lightning)
944	Dynamite; Nitrocotton, Gelatin		0/0	Dry House	Fire-Explosion	500/-	Open flames (from range fire)
1015	Ammonium Nitrate (oil sensitized)			Oven Operation	Fire		Thermal (thermostat failed)
1029	XM-30 Sustainer Motors (4)	9200	0/11	Curing Oven	Explosion		1) Thermal (leaking propellant into hot electric heating coils) 2) Friction/impact (from mechanical failure of clamps, therefore dropping the motors)

SUMMARY OF SELECTED ACCIDENTS WHICH OCCURRED DURING DRYING (cont)

ASESB NO.	AGENT	AMOUNT (LB)	FATALITIES/ INJURIES	COMPONENT OR OPERATION	OUTPUT-TYPE	MAX. DISTANCE MISSILE/GLASS BREAKAGE (FT.)	PROBABLE CAUSES
1034	Explosive Comp Slurry (Bismuth Trioxide, Magnesium, 1½% Binder Base-Methyl Ethyl Ketone (MEK)			Curing Oven	Explosion		Thermal: 1) Exothermic reaction of fuel 2) Hot spot formation 3) Organic peroxide from breakdown of MEK could have contributed in heat generation
1311	Arcite Terrier Sustainer grain		0/0	Curing Operation	Explosion-Fire		Thermal: contamination of mold with iron rust
386-7	Picric Acid			Drying Facility	Fire		Impact between bogie and iron pipe
386-8	Picric Acid			Picric Acid Drying Stove	Fire		Friction-spark ignition due to metal surface abrasion
386-9	Picric Acid			Drying Room	Fire		Friction between steam pipe and the support
386-12	Picric Acid			Stove Bed	Fire		Friction-metal to metal
386-18a	Picric Acid			Drying Stove	Fire		Thermal-hot spot on steam pipe
386-18b	Picric Acid			Drying Stove	Fire		Thermal-hot spot on steam pipe
386-18c	Picric Acid			Drying Stove	Fire		Thermal-hot spot on steam pipe
386-18d	Picric Acid			Drying Stove	Fire		Thermal-hot spot on steam pipe
386-25	Picric Acid			Drying Room	Fire		Unknown
386-27	Picric Acid			Drying Shed	Fire-Explosion		Unknown
374	Cordite/ Acetone Alcohol	55,790	2/10	Recovery Stove/ Drying Process	Explosion-Fire		No specification
1033	Dry Nitro-starch	100	1/0	Unloading Dryer	Explosion	75/0	Unknown
1287	Ball Powder WC852	6000 (initial) 13,780	0/0	Dryer	Fire-L.O. Detonation	Building area	Excessive heat buildup

SUMMARY OF SELECTED ACCIDENTS WHICH OCCURRED DURING DRYING (cont)

ASESB NO.	AGENT	AMOUNT (LB)	FATALITIES/ INJURIES	COMPONENT OR OPERATION	OUTPUT-TYPE	MAX. DISTANCE MISSILE/GLASS BREAKAGE (FT.)	PROBABLE CAUSES
1467	Black powder boron pot. nitrate tungsten delay mix	20 20 1000	0/0	Dryhouse and oven	Explosion		Unknown
910	Dry Nitro-starch	850	0/0	Dryer	Explosion	75/-	Overheated motor in fanhouse
750	Nitrocotton	700	0/0	Dryhouse	Explosion	100/-	Unknown
712	Nitrocottom	400	1/0	Dryhouse	Explosion	1200/200	Unknown
394	TNT	300 stove 1000 drying 150 hopper 5-6 tons stored	0/0	Vacuum Drying Stove	Explosion		1) Decomposition accelerated by pressing of ammonium nitrate due to contamination in drying stove
393a	Cordite RDB		0/0	Stove	Fire		Unknown
393b	Cordite RDB		1/0	Stove	Fire		Ignition of solvent vapors
393c	Cordite RDB		2/2	Stove	Fire		Ignition of inflammable vapor in the vapor piping
393d	Cordite MD		1/5	Recovery stove	Fire		Vapor ignition of acetone air mixtures which were to be recovered
393e	Cordite RDB		0/1	Stove	Fire		Ignition of vapors
376	Di-nitro-phenol-picric acid		7/69	Drying room	Fire-Explosion	15 yd center 12 ft deep 500 yd missile	Initiation-smoking
383	Cordite RDB	47,332	0/0	Recovery stove	Explosion		Vapor ignition
534	Fulminate and tetraethylic substance			Hot air stove	Explosion		Overheating
1123	Mercury fulminate	55	2/0	Dehydration	Explosion	Building area	1) Initial ignition of dry fulmate by friction (metal-metal) with subsequent ignition of alcohol-air mixture in collector)

SUMMARY OF SELECTED ACCIDENTS WHICH OCCURRED DURING DRYING (concl)

ASESB NO.	AGENT	AMOUNT (LB)	FATALITIES/ INJURIES	COMPONENT OR OPERATION	OUTPUT-TYPE	MAX. DISTANCE MISSILE/GLASS BREAKAGE (FT.)	PROBABLE CAUSES
821	Single base prop (shock gel process)		0/1	Rotating drum dryer	Explosion		1) Loose metal supports within dryer initiated pellets by friction 2) Impact
1045	Mercury fulminate	5	0/0	Drying Operation	Explosion	60/50	Unknown
1125	Lead Azide Lead trinitro resorsonate	27	1/0	Dry house	Explosion	chamber/building	Impact (?)
1129	Lead Azide Lead Scyphnate	22	1/0	Drying Operation	Explosion-Fire	-/60	Impact
1202	HP-2 Experimental prop	10 oz		Cure oven	Explosion	Immediate oven area	Thermal instability between HP-2 and other ingredients
1107	EM-27 prop			Curing facility	Explosion		a) High temperature b) Equipment failure c) Impact (slippage)
1056	FOC experimental motor mold			Curing house	Explosion-Fire	Immediate bay area	Impact and sympathetic reaction
349	Fire gunpowder	2800	5/2+	Dry house (handling)	Explosion	350/-	ESD-explosive dust explosion

SUMMARY OF SELECTED ACCIDENTS WHICH OCCURRED IN REACTORS

ASESB NO.	AGENT	AMOUNT (LB)	FATALITIES/ INJURIES	COMPONENT OR OPERATION	OUTPUT-TYPE	MAX. DISTANCE MISSILE/GLASS BREAKAGE (FT.)	PROBABLE CAUSES
1476	TNT	10,000	0/6 major 100 minor	Nitrator-separator	Explosion	3000/ 70 ft crater	Thermal/exothermic instability-inadequate mixing due to hose obstruction with agitator
1267	Nitro-glycerine	--	--	Nitrator	Explosion	--	Chemical decomposition of old spent acid within reactor-exothermic
502	TNT	--	0/0	Tri-nitrator	Fire	--	Thermal instability-caused by operator procedure
1117	Hexogene	--	0/0	Nitrator	Reaction chemical	--	Thermal instability-caused by mach failure
1191	TNT	--	0/3	Dinitrator	Reaction chemical violent	--	Exothermic reaction
1111	Nitro-glycerine	Small		Nitrator	Explosion	--	Temperature increase
982	Tetranitro-methane	--	--	Reactor	Explosion	Building destroyed	Poor agitation caused a runaway chemical reaction with excessive heat build-up; mechanical failure of agitator (friction/ impact)
1119	Nitro-glycerine	1100	2/-	Nitrator-	Explosion	500 ft building destroyed	Block cock - friction oriented
380	Nitro-glycerine	5500	--	Nitrator-separator	Flame-explosion	--	1) Decomposition of impurities adhering to reactor shell 2) Decomposition of flaeculent matter on surface of separated nitroglycerine

SUMMARY OF SELECTED ACCIDENTS WHICH OCCURRED IN REACTORS (cont)

ASESB NO.	AGENT	AMOUNT (LB)	FATALITIES/ INJURIES	COMPONENT OR OPERATION	OUTPUT-TYPE	MAX. DISTANCE MISSILE/GLASS BREAKAGE (FT.)	PROBABLE CAUSES
908	Nitro-glycerine		0/0	Dinitrator waste acid	Explosion	--	Contamination during cleaning exercise + thermal/decomposition
312	Nitro-glycerine	450x3 1350 lb	2/0	Reactor	Explosion	Building destroyed ~500'	Unknown
603	Nitro-glycerine	--	0/1	Nitrator (Biazzi system)	Explosion	~1000' plant destroyed	Decomposition of nitro-glycerine-runaway reaction due to excessive acid addition; heat generated and fast temperature rise
907	PETN Petrin acrylate (rocket propellant)	~1 lb	1/3	Nitrator	Explosion	~100 ft	Instability of impure PETN included with acid at ambient temperature and further decomposition of explosion with the addition or a water-acid base (accidental addition of water)

SUMMARY OF SELECTED ACCIDENTS WHICH OCCURRED IN REACTORS (cont)

ASESB NO.	AGENT	AMOUNT (LB)	FATALITIES/ INJURIES	COMPONENT OR OPERATION	OUTPUT-TYPE	MAX. DISTANCE MISSILE/GLASS BREAKAGE (FT.)	PROBABLE CAUSES
368	TNT		0/0	TNT nitration process	Fire		1) Insufficient agitation-thermo-chemical runaway reaction 2) Chemical decomposition
373	DNT-TNT-acid mix		0/0	Nitrating reactor	Fire		1) Chemical ignition (acid-covered combustible) 2) Agitation failed - unstable temperature gradient
389	Nitroglycerine	6500	2/0	Nitrator	Explosion		1) Spontaneous decomposition 2) Friction - tool
392	Picric Acid		5/-	Nitration-waste water recovery	Explosion-fire		1) Chemical reaction (picric acid + iron oxide); sudden heat addition
462	Nitroglycerine	Initial 7000 lbs	3/0	Nitrator	Explosion	700/1200 -3100	Unknown, not specified-guess impact of foreign particle
498	Bi-oil + Acid (aulpheric nitric)	500-800		Trinitrator process	Explosion	200-1390 250-300	Thermochemical (extreme temperature rise caused by rapid addition of bi-oil and acid)
501	TNT		0/1	Trinitration process	Fire-explosion		Exothermic instability-runaway reaction insufficient cooling and rapid addition of bi-oil
678	TNT (oleum acid bi-oil)		0/0	Trinitrator Operation	Explosion		Extreme exothermic reaction due to insufficient agitation and overheat
1466	TNT		0/2	Trinitration	Explosion-fire	300/-	Unknown - no specification
1566	Nitroglycerine		0/0	Batch NG nitrator	Explosion	-/3-5 miles	Unknown - no specification

SUMMARY OF SELECTED ACCIDENTS WHICH OCCURRED IN REACTORS (concl)

ASESB NO	AGENT	AMOUNT (LB)	FATALITIES/ INJURIES	COMPONENT OR OPERATION	OUTPUT-TYPE	MAX. DISTANCE MISSILE/GLASS BREAKAGE (FT.)	PROBABLE CAUSES
1573	Nitro-glycerine		4/0	Nitration facility	Explosion		Human error - no specification on stimulus
1172	NFPA-NFPOR (Classified)		0/0	Nitration reactor			Thermal: Unstable exothermic release due to lack of agitation and cooling
1183	Sulfuric Nitric acid plus toluiene bi-oil		1/3	Nitration process	Violent Reaction		Thermal: spontaneous heat addition; rapid volatilization and explosion
1236	DNT	6000		Tri-nitrator	Fire-Explosion		Thermal: high heat content with insufficient cooling (no compliance with SOP)
1621	Nitro-glycerine		0/0	Continuous nitrator process	Explosion		Unknown - no specification
1683	6-Amino-pencillanic acid, S-oxide, trimeric acetone peroxide			Small reactor	Explosion		1) ESD 2) Friction (technician touched filter cake with steel spatula For trimeric acetone peroxide sensitivity is: 11.5 mJ electric spark, impact-2 Kg at 10 cm, friction-.5 Kg weight
161	TNT			Nitrator	Explosion-Fire		1) Thermal (no agitation) 2) Chemical Decomposition of nitrobody
386-14	Picric Acid			Nitration	Fire		Thermochemical reaction with organic
386-15	Picric Acid			Nitrating house	Fire		Thermochemical reaction with organic

SUMMARY OF SELECTED ACCIDENTS WHICH OCCURRED DURING SOLVENT RECOVERY AND IN ACID CONCENTRATORS

ASESB NO.	AGENT	AMOUNT (LB)	FATALITIES/ INJURIES	COMPONENT OR OPERATION	OUTPUT-TYPE	MAX. DISTANCE MISSILE/GLASS BREAKAGE	PROBABLE CAUSES
150	Smokeless Powder	296-550	49/110	Solvent Recovery Line	Fire-Explosion	-3/4 mile	Nine (9) possibilities listed
234	Smokeless Powder		5/0	Solvent Recovery Process/Chute	Explosion		1) ESD 2) Friction-tumbling
954	Nitrobody/ Sulphuric Acid & Oil			Concentrator/ Recovery of Sulphuric acid	Flash Fire		1) Electrical short 2) Contamination
386-24	Picric Acid			Lead Pan Concentrator	Explosion		Unknown, acid overflow
386-26	Picric Acid			Acid Concentrator Plant	Explosion		Unknown, acid leak
386-29	Picric Acid			Acid Concentration House	Explosion		Unknown

SUMMARY OF SELECTED ACCIDENTS WHICH OCCURRED DURING WASHING OPERATIONS

ASESB NO.	AGENT	AMOUNT (LB)	FATALITIES/ INJURIES	COMPONENT OR OPERATION	OUTPUT-TYPE	MAX. DISTANCE MISSILE/GLASS BREAKAGE (FT)	PROBABLE CAUSE
375	TNT	4-1/2 tons (nearby)		Wash house/ scaling house	Flame-Explosion		Spontaneous ignition 1) TNT dust in fume pipe 2) bearings of fan-hot spot 3) heating pipe 4) decomposition of TNT by alkali
682	ANM50XA3 containing detonator and tetryl explosive incendiary bomb		1/16	Washing operation on bomb?	Explosion		Unknown, guess-defective fuze
?	NOS Indianhead Maryland 13, Sept. 1971 (nitroglycerine)	450	2/0	Prewash tank	Explosion		Unknown, no specification
382	Nitroglycerine Nitrocotton	3578 936	6/1	Washing	Explosion		Friction-wooden clip and rubber tube
1189	Lead Azide (alcohol and freon)	2	1/-	Filling flask Washing-aspiration	Explosion		Friction due to bumping of flask and funnel

APPENDIX C

JUSTIFICATION FOR CHOOSING SPECIFIC SENSITIVITY TESTS FOR EACH PROCESS OPERATION

JUSTIFICATIONS FOR CHOOSING SPECIFIC SENSITIVITY TESTS FOR EACH OF THE DIFFERENT PROCESS OPERATIONS

Belt Conveyors

1. Impact
   - on layer or individual particles, whichever is appropriate
   - from dropped item (e.g., tool) or mechanical failure of machinery
2. Impingement
   - during loading and emptying, dropping particles onto belt or into next container
3. Rubbing Friction
   - powder on slipping rollers, etc.
   - person pushing article across apparently empty belt
4. ESD

   Dot determination
   - removing particles at exit end of belt (very unlikely scenario -- omit)

   Bed/layer ignition (if vapor is present evaluate ignition there too)
   - discharge from ungrounded person
5. Thermal

   Local hot spots
   - welders spark
   - cigarette cinder
   - fire brand (secondary event)
   - hot metal fragment from part failure

   Autoignition
   - heat generated from stuck roller, failed bearing, belt slippage, motor burnout, etc.
6. Compression/Pinch
   - material caught between belt and roller
   - material gets into moving parts (falls under category of weak, slow impact)

Screw Conveyors

1. Impact

   Within bulk
   - machine failure causes impacts internal (weak)

on layer or particle

- human impacts (dropped part/hitting bolt)during maintenance, cleanup

2. Impingement
   - during loading/emptying of machinery
3. Rubbing friction
   - clump or individual particle gets caught between screw tube wall
4. ESD

   Ego determination
   - charge buildup as powder is rubbed on screw tube wall

   Bed/layer ignition
   - breakdown within powder bed
   - discharge from ungrounded person to dust present on outside of tube or at entrance or exit hoppers
5. Thermal

   Local hotspots
   - welders spark, cigarette, fire brand at entrance or exit hoppers
   - hot metal chip from part failure

   Autoignition
   - [illegible] metal shaft transmitting heat from motor burnout
6. Compression/Pinch
   - particles pressed in machine parts (falls under category of weak, slow impact)

Bucket Conveyors

1. Impact

   On layer or individual particles, whichever is proper
   - from dropped item or mechanical failure of equipment
2. Impingement
   - on filling or emptying buckets
3. Rubbing Friction
   - person scraping to clean out bucket
   - powder gets caught in moving parts
4. ESD bed/layer ignition (if vapor is present, evaluate vapor ignition also)
   - discharge from ungrounded person
5. Thermal

Local hot spot

- welders spark, cigarette, firebrand

Autoignition

- dust/powder gets on hot motor, failed bearing, etc.

6. Compression/Pinch
   - powder gets into moving machine parts (falls under category of weak, slow impact)

Pneumatic Conveyors, Jet Mill, Air Mixer, Cyclone, Dust Collector, etc.

1. Impact, on layer
   - person chipping off scale on inside equipment walls
   - dropped item during maintenance
2. Impingement
   - particle-wall, particle-particle impacts during operation
3. ESD particle cloud ignition
   - discharge within material cloud inside equipment
     - from charging with cloud (not likely to be strong)
     - from charged ungrounded equipment part inside item
4. Thermal

   Local hot spot
   - incendiary spark produced by foreign piece of material which got into the system

   Autoignition Temperature
   - dust leaks and gets on blower motor (very weak argument -- omit)
5. Compression/Pinch
   - dust gets into blower moving parts (very weak argement)

Hoppers

1. Impact

   Within Bulk
   - butterfly valve at exit closes on individual particles/ layer (whichever is proper)
   - item dropped into hopper
   - chipping out residue during cleanup
   - dropped cover
   - person hits bolt to loosen
2. Impingement

- particles hit wall on filling

3. Rubbing Friction
   - person scraping out residue during cleanup
4. ESD

   ESD determination
   - bed charging upon emptying

   Bed/layer ignition (consider vapor if present)
   - within sliding bed (not likely)
   - from ungrounded person

   Cloud ignition
   - discharge from lip of entrance duct upon filling
5. Thermal

   Local Hot Spot (Open Hopper Only)
   - welding, smoking firebrand

   Autoignition
   - dust gets on hot motor, etc., in vicinity (weak argement -- omit)

Tote Bins

1. Impact

   Within Bulk
   - closing of exit valve (weak argument)

   On layer or individual particle
   - item dropped into bin
2. Impingement
   - particles falling into bin on filling
3. Intermediate Scale Impact
   - bin gets loose from operator and rams into another equipment item
4. Rubbing Friction
   - person scraping out residue on cleaning
   - dust on floor stepped on or slid on by bin wheel
5. ESD

   ESD determination
   - bed charging upon emptying bin

   Bed/layer ignition
   - within sliding bed
   - from ungrounded person

Cloud/vapor ignition

- ungrounded bin discharge to filling/emptying flexible connection upon filling/emptying

6. Thermal

Local hot spots

- welding, smoking, firebrand for open bin

Autoignition

- dust gets on hot motor, etc. (weak argement --omit)

Screening

1. Impact

On layer or individual particles (whichever is appropriate)

- dropped item
- equipment failure (e.g., shaker linkage)

2. Impingement

- particles falling from a screen level to the next level or onto the bottom surface
- particles falling onto a screen upon filling

3. Rubbing friction

- powder getting caught in moving screen (e.g., between wires)
- person scraping wall during cleaning

4. ESD

Bed/layer ignition and cloud ignition (vapor if proper)

- discharge from person upon cleaning
- discharge from ungrounded screen or partly insulated screen wires

5. Thermal

Autoignition

- dust gets on hot shaker motor external
- motor burnout heat conducts into screen parts

Pressing

1. Impact

On layer or individual particle (whichever proper)

- failure of machine parts
- dropped item during maintenance
- person trying to pry/knock out stuck cake

2. Impingement
   - particles entering mold during filling
3. Viscous Friction
   - extrusion of material around edge of press with poorly fitting mold
     (design and special safety problem)
4. ESD bed/layer ignition (consider vapor if exists)
   - discharge from ungrounded person
5. Thermal

   Local hot spot/autoignition (depending on size of foreign part)
   - foreign metal piece deformed in mold or shears a chip off the mold wall
   - welding, smoking, firebrand
     (weak arguments)
6. Compression/Pinch
   - over design max Pressure (P) or $\frac{dP}{dt}$
     (design and special safety problem)
7. Intermediate Scale Impact
   - cake dropped
8. Rubbing Friction
   - friction during cake removal

Extrusion

1. Impact

   On layer or individual strand (whatever is appropriate)
   - due to equipment failure
   - due to person dropping item onto strand, etc.
2. Viscous Friction
   - overdesign extrusion
   - extrusion with foreign piece
3. Rubbing Friction
   - friction as strand or sheet moves through die
   - person steps on strand and slides
4. ESD bed/layer ignition
   - discharge from ungrounded person

   ESD [illegible] determination
   - charge buildups on strand as it passes through die
     (discharge on surface back to die lip)
     (weak argument -- omit)

5. Thermal

Local hot spot

- foreign part pushed through die
- welding, smoking, firebrand

Autoignition

- ignition to heating of foreign material in die (design and special safety problem)

6. Compression/Pinch

- compression of material in die (design and special safety problem)

Mills

1. Impact

On layer or individual particle

- machine part failure
- tooth impacts on chunks

2. Impingement

- particle impacts on teeth
- particles dropped onto the machine on filling

3. Intermediate Scale Impact

- hammer mill hitting chunk of material

4. Rubbing Friction

- powder gets into bearing, moving parts
- scraping wall on cleaning
- chunk rubbed between tooth and wall

5. ESD

Cloud ignition (not likely)

- discharge of particle cloud within cloud
- ungrounded cutter discharges in cloud

6. Thermal

Local hot spot

- foreign particle enters (incendiary spark)

Autoignition

- hot shaft heated by motor burnout or bearing failure

7. Compression/Pinch

- powder gets into moving parts (e.g., bearing) and is crushed there (handle as slow, weak impact)

Glazing, Coating and Batch Drum Operations

1. Impact

   Within bulk, on layer or particle

   - foreign part (e.g., tool) tumbles inside drum
   - person drops item into drum
   - slamming drum door closed

2. Impingement
   - particles dropped into drum
   - particles tumbling in drum

3. Rubbing Friction
   - person cleaning out (scraping) drum
   - powder caught in drum shaft bearings
   - opening/closing door in drum

4. ESD

   ESD determination

   - bed charging of powder sliding in drum (weak argement -- omit)

   Bed layer ignition

   - ignition within charged bed
   - from ungrounded deflector plate/etc. inside drum
   - from person discharge

   Cloud ignition

   - from ungrounded part inside drum (e.g., foreign metal piece)

5. Thermal

   Local hot spot (open drum only)

   - welding, smoking, firebrands, etc.

   Autoignition

   - Operation at overdesign temperature

Dryer

1. Impact

   On individual particles or layer (which is proper)

   - person drops item onto layer on belt

2. Impingement
   - particles hitting surface on filling or emptying

3. Rubbing Friction

- powder/dust getting between belt/rollers, in bearings, etc.

4. ESD layer ignition (consider vapor of present)
   - ungrounded person
5. Thermal

   Local hot spot
   - welding, smoking, firebrand
   - hot chip from part failure

   Autoignition
   - over design drying temperature

   Intense Radiant Heating (specialized)
   - some systems were at one time suggested using intense radiant heating to vaporize liquid in drying
6. Compression/Pinch
   - dust/powder pinched between belt/rollers or gets in moving parts
     (handle as slow, weak impact)

Melt Pour, Casting

1. Impact

   Within bulk
   - agitator impact

   On layer
   - dropped item on liquid
   - person chipping at residue on container wall during cleaning
2. Intermediate Scale Impact
   - melt kettle dropped or hit another item during pour operation
3. Rubbing Friction
   - scraping at kettle wall during cleaning
   - agitator rubs build up residue on container wall
4. ESD bed/layer ignition (consider vapor if there)
   - ungrounded person
   - ungrounded kettle
5. Thermal

   Local hot spot
   - welding, smoking, firebrand

   Autoignition/Runaway chemical reaction
   - overdesign kettle temperature

Chutes

1. Impact

   On layer or individual particles

   - dropped item

2. Impingement

   - particles entering or exiting
   - particle-particle/particle wall impacts during travel

3. Rubbing friction

   - during cleaning, scraping residue off wall

4. ESD

   $\varepsilon\sigma\tau$ determination

   - bed charging

   Bed/layer, cloud, vapor ignition

   - ungrounded person
   - discharge within bed

5. Thermal Local Hot Spot

   - welding, smoking, firebrands

   Autoignition

   - dust gets on other nearby equipment that is hot, e.g., motor (somewhat weak argument)

Reactors/Wash, Mix, Hold Tanks

1. Impact

   Within Bulk

   - Agitator impact

   On layer (somewhat weak arguments)

   - dropped cover
   - impact on residue on outside of vessel

2. Rubbing Friction

   - Agitator scrapes on builtup layer of residue/scale

3. ESD Vapor Ignition (if vapor is present)

   - discharge from ungrounded person

4. Thermal

   - Runaway chemical reaction due to loss of cooling, loss of mixing, leak in heat exchanger, operation at overdesign T, etc. (design and special safety problem)

Gravity Separators

1. Impact (on layer)
   - dropped item
   - person trying to unclog chute to next vessel
2. ESD

   εσr determination
   - charge separation on separating phases

   Bed/layer ignition (consider vapor of present)
   - at interface between phases
   - ungrounded person

   Rubbing friction
   - person trying to unclog chute to next vessel
3. Thermal

   Autoignition
   - operating overdesign temperature

Centrifugal Separators

1. Impact

   Within bulk
   - foreign part enters and is thrown to outer wall
2. Rubbing friction
   - residue builds up on moving parts or bearings
3. ESD

   εσr determination
   - charge separation with phase separation

   layer ignition
   - due to charge separation
4. Thermal

   Autoignition/runaway
   - operation over temperature

Filter

1. Impact on layer or individual particle
   - dropped item
2. Rubbing Friction
   - material gets into moving parts of system (e.g., between belt/roller)

- friction at belt scraper, especially with foreign piece caught there

3. ESD

   Bed/layer ignition (vapor if present)

   - ungrounded person

4. Thermal

   Local hot spot

   - welding, smoking, firebrand

   Autoignition

   - overdesign operating temperature
   - heating via failed bearing or burnout motor

5. Compression/Pinch

   - material on moving machine parts, e.g., between belt/roller
     (handle as slow, weak impact)

Flaker Drum, Belt Flaker

1. Impact

   On layer or individual particle (both are present here)

   - dropper item
   - foreign part falls into product container with flakes

2. Impingement

   - flakes falling into product container

3. Rubbing friction

   - powder/dust gets into moving parts
   - friction at belt scraper

4. ESD bed/layer ignition (vapor if present)

   - ungrounded person

5. Thermal

   Local hot spot

   - welding, smoking, firebrand

   Autoignition

   - heating via failed bearing, burned out motor, etc.

6. Compression/Pinch

   - powder/flakes get pinched in moving machine parts, e.g. between belt/roller
     (handle as slow, weak impact)

Product Pumps

1. Impact

   In bulk

   - part failure or foreign material

2. Adiabatic Compression

   - compression of a liquid air bubble passing through pump

3. Rubbing Friction

   - friction of impellar on builtup wall scale

4. Viscous Friction

   - shear flow of liquid through pump

5. Thermal

   Autoignition

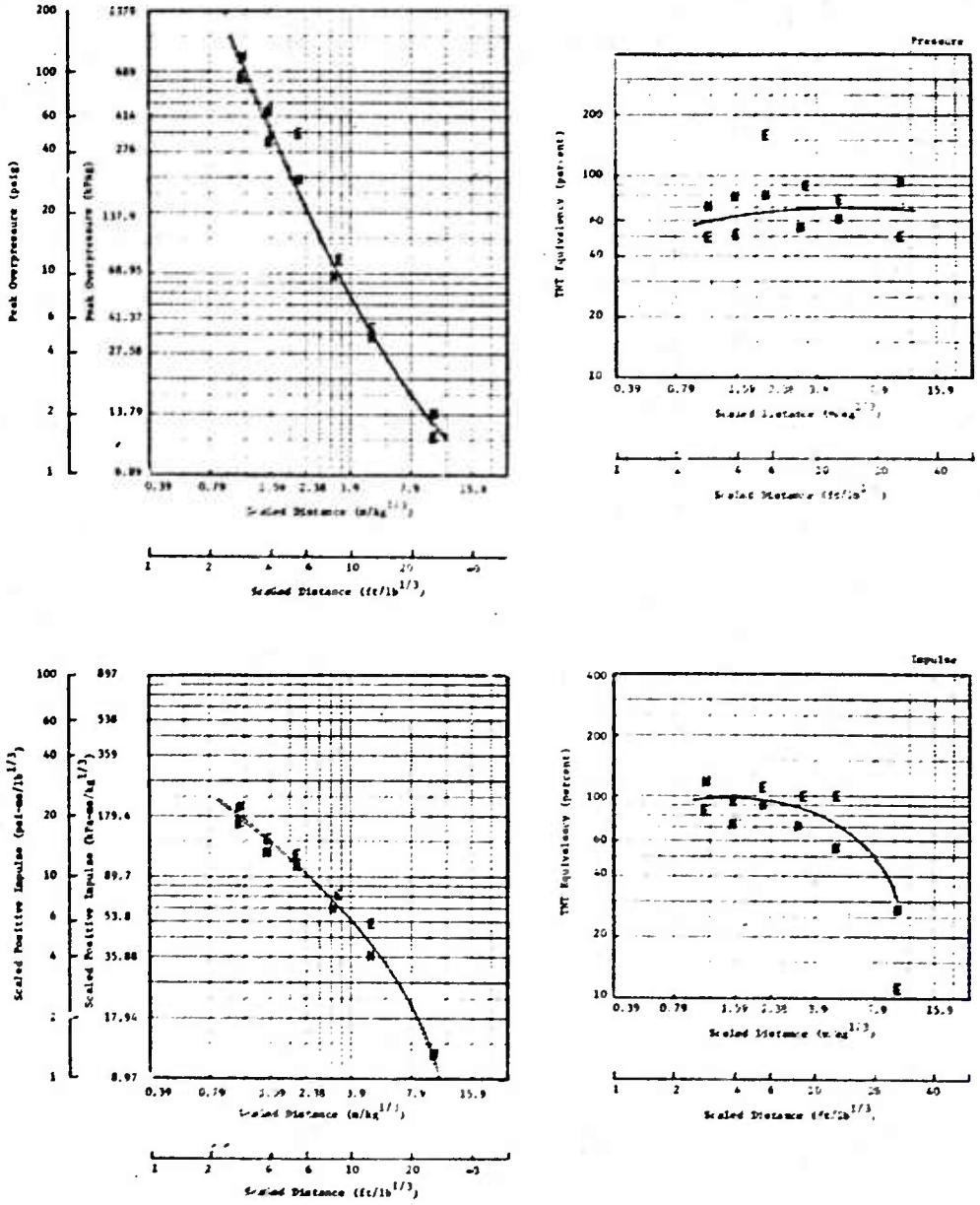
   - overtemp design
   - motor burnout
   - bearing failure

6. Compression/Pinch

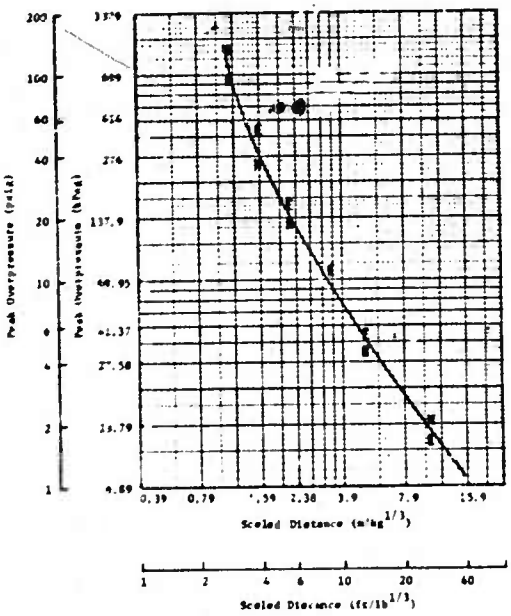
   - overdesign pressure (design problem)

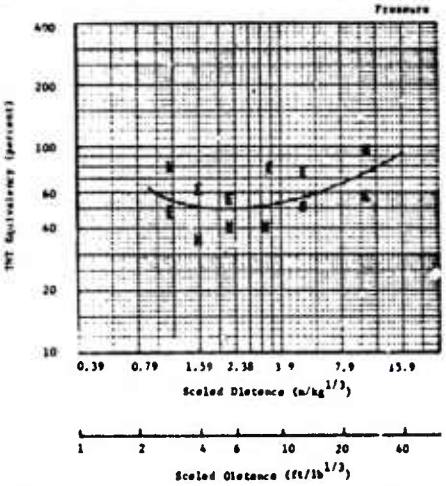
APPENDIX D

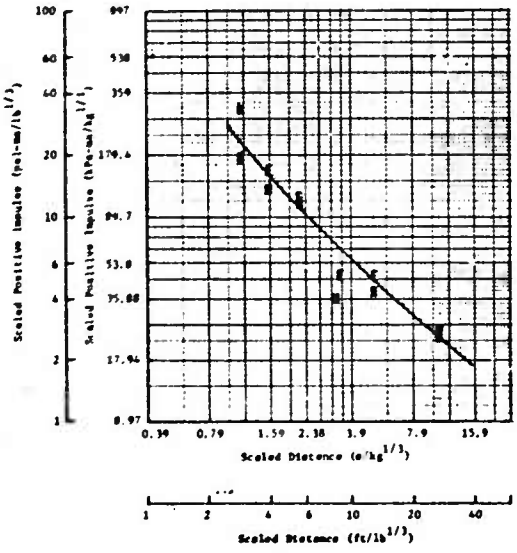
AIRBLAST TEST RESULTS

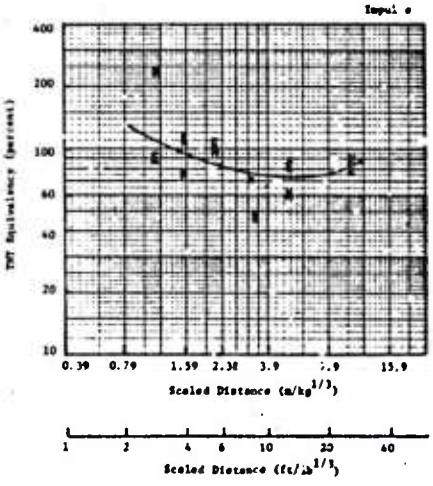


Airblast output for test 1 (M26)









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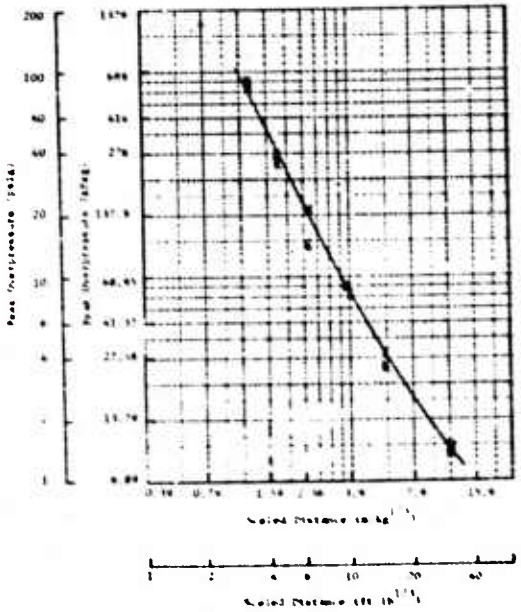

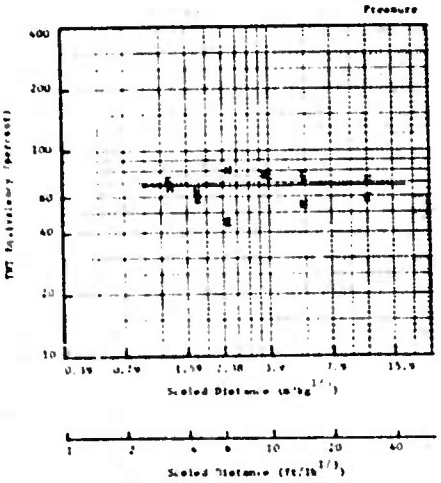

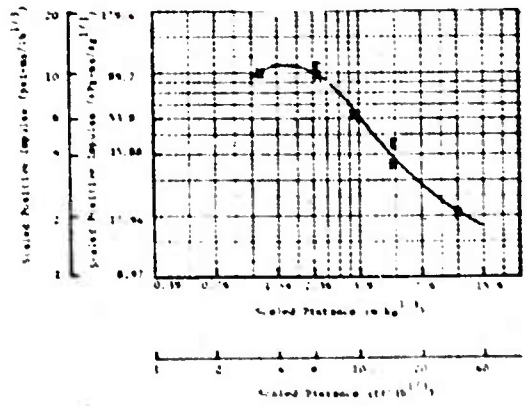

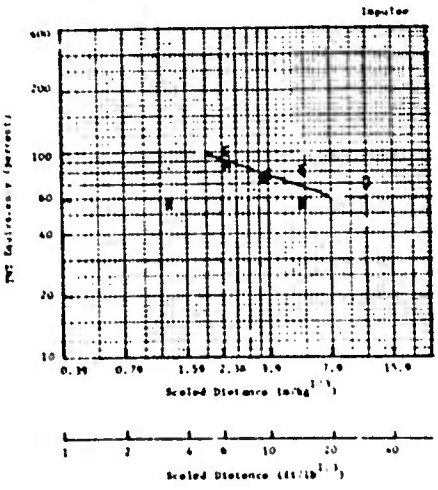

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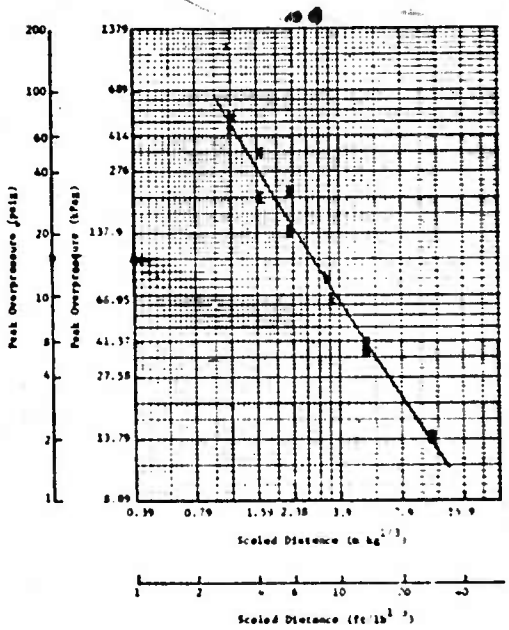

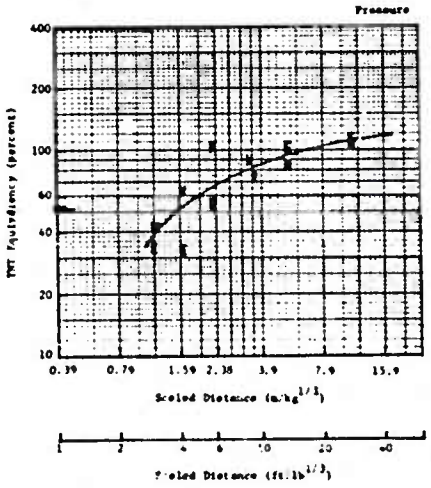

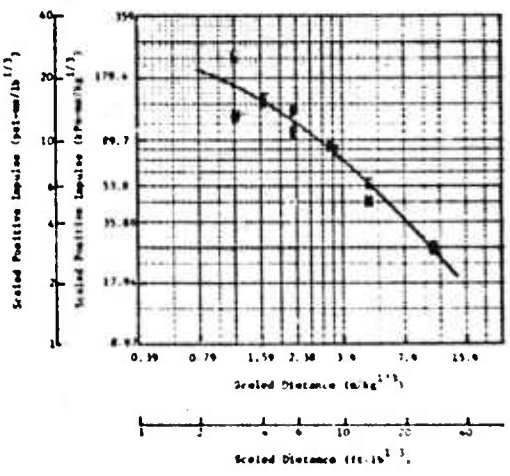

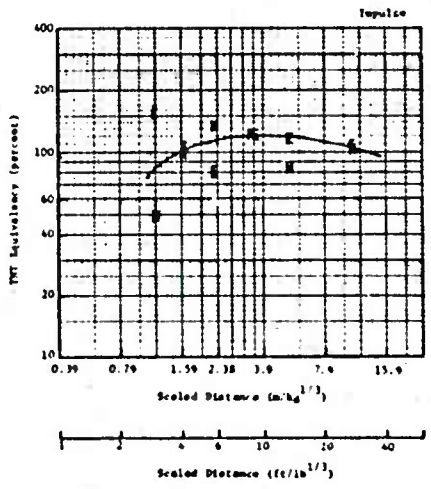

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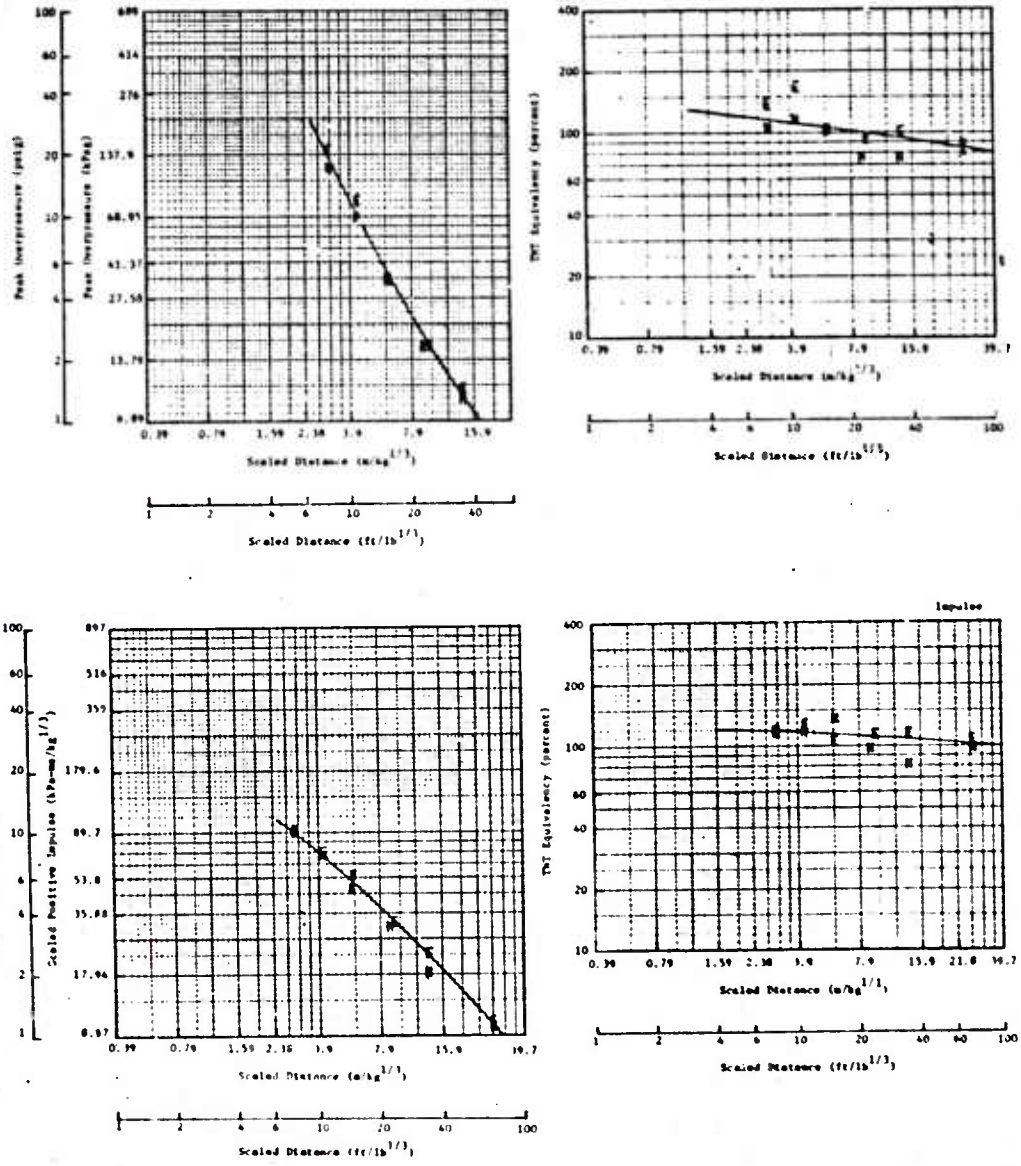

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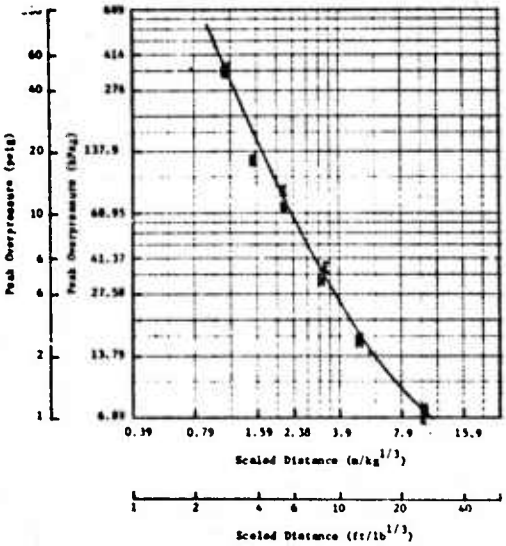
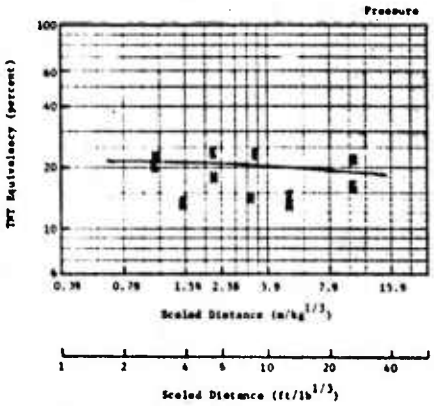
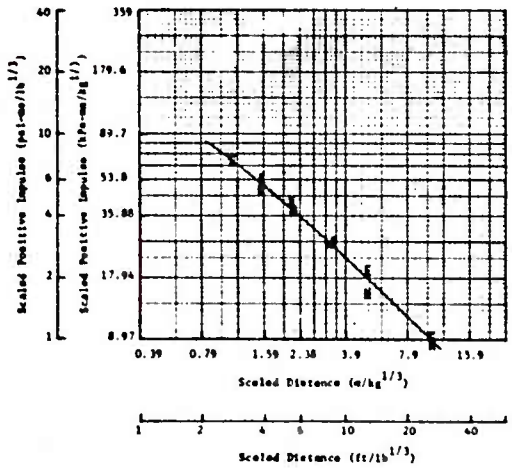
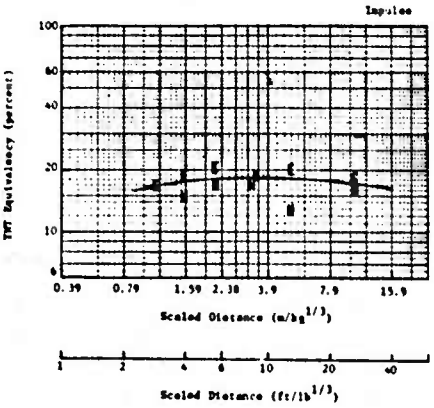
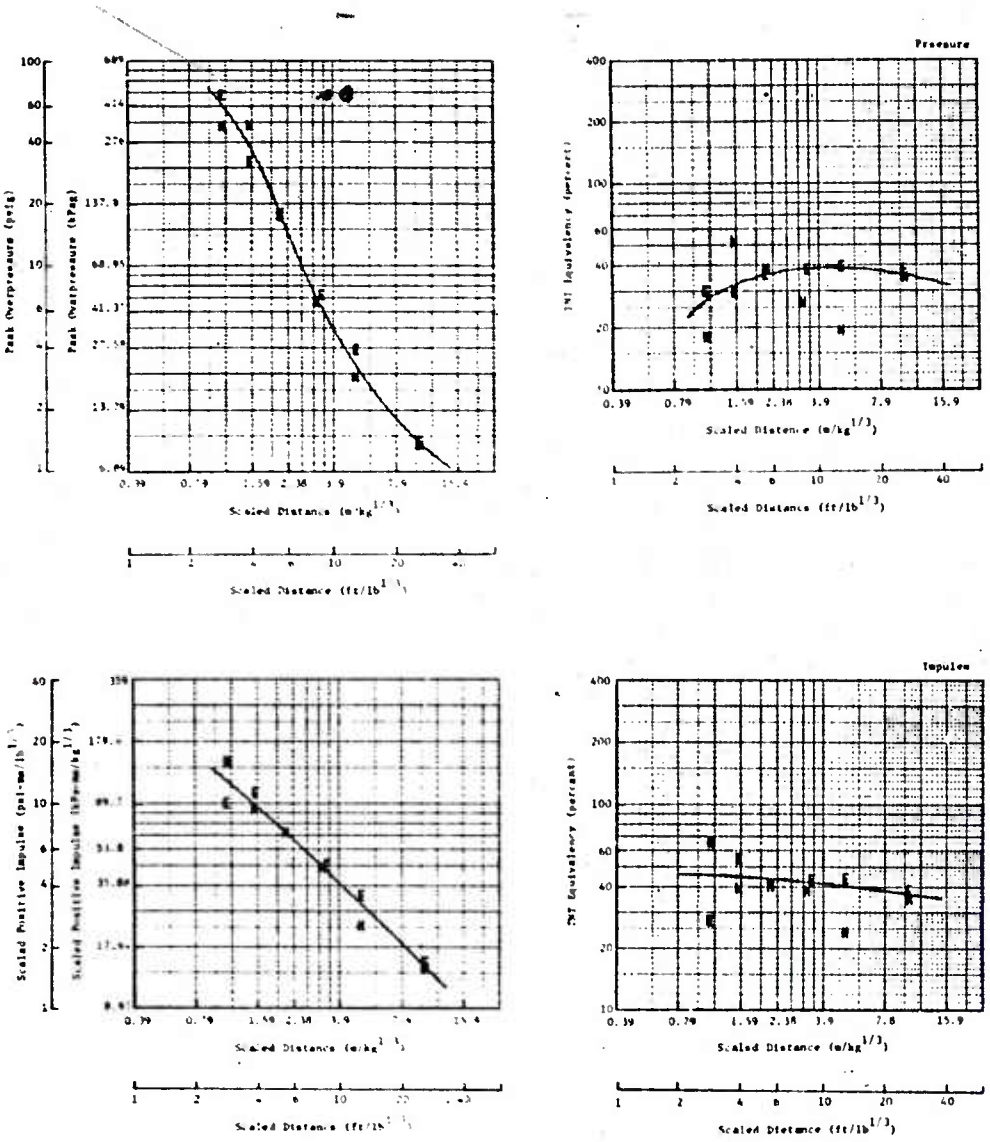








Airblast output for test 7 (M30)



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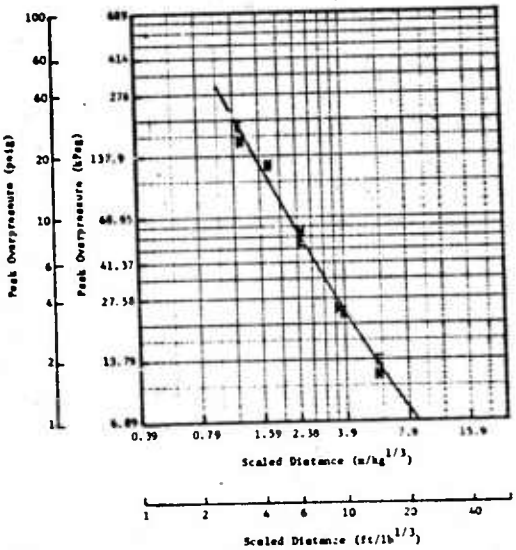
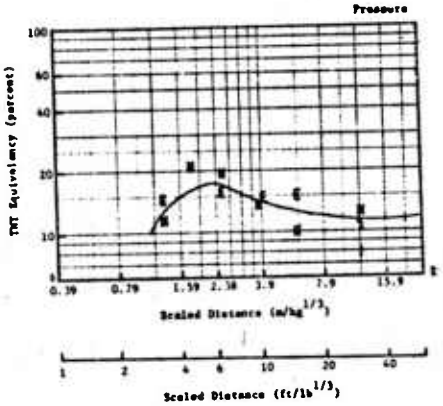
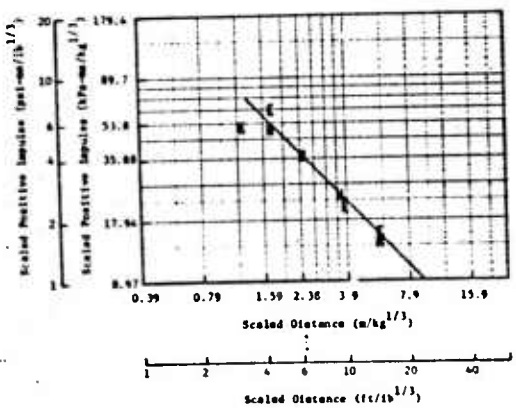
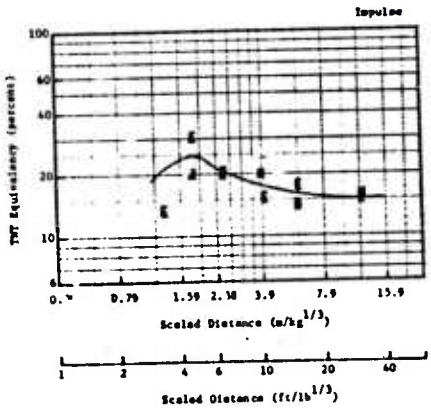
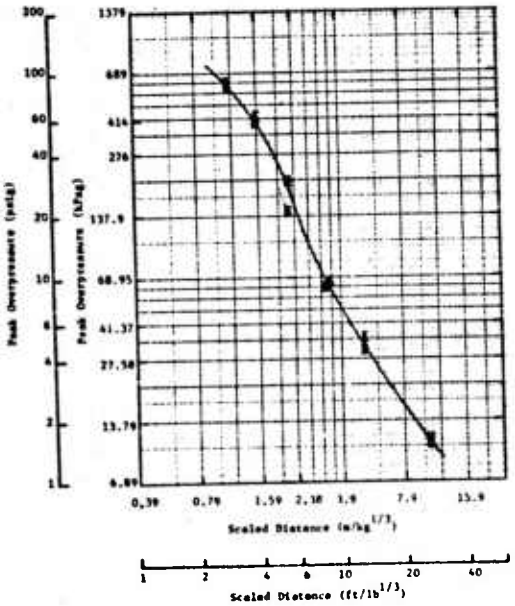


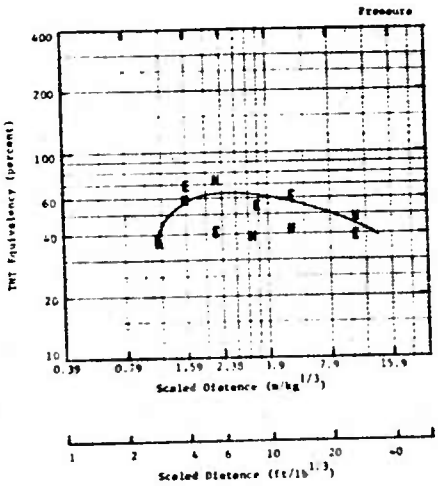


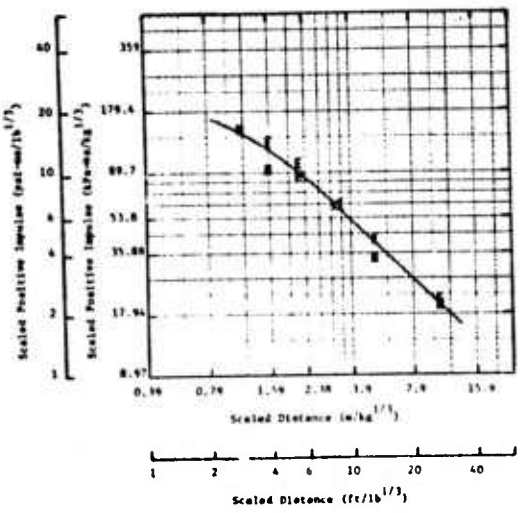


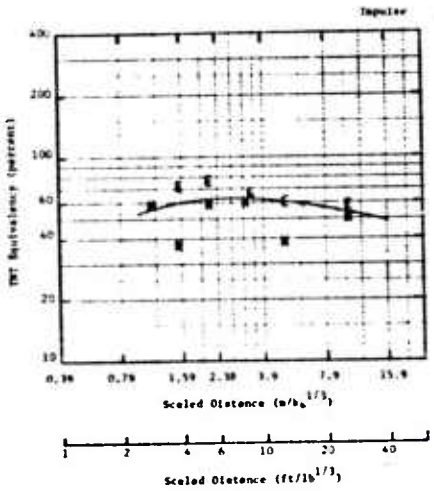


Airblast output for test 9 (M30)









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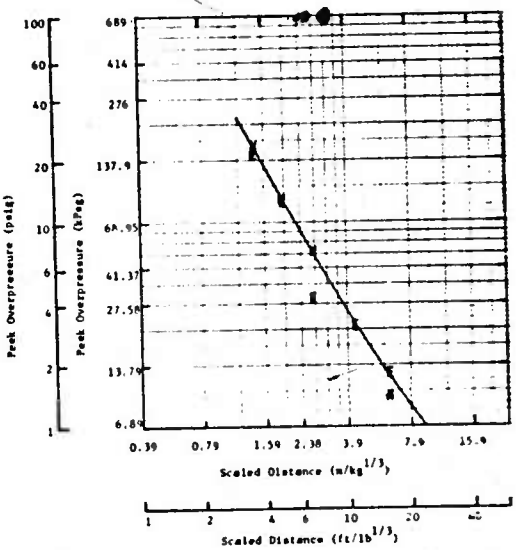
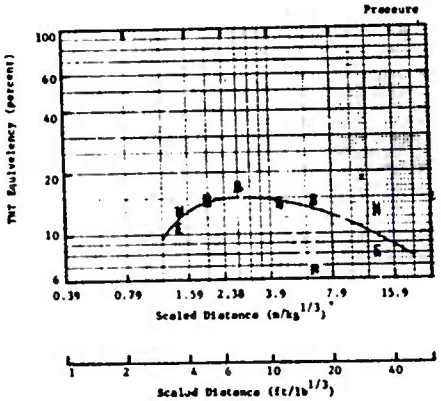
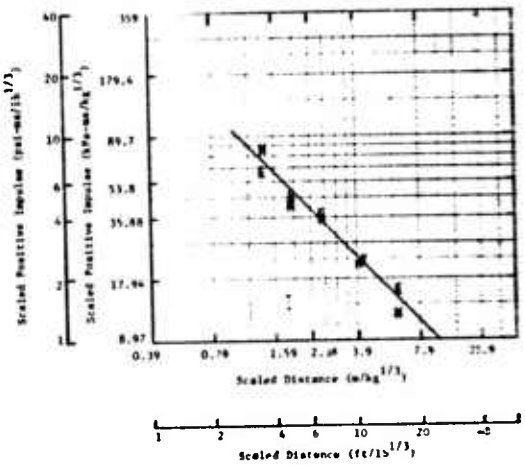
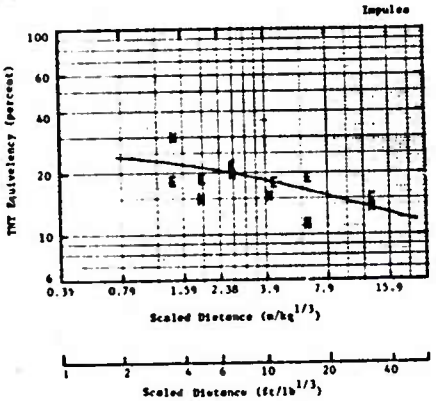
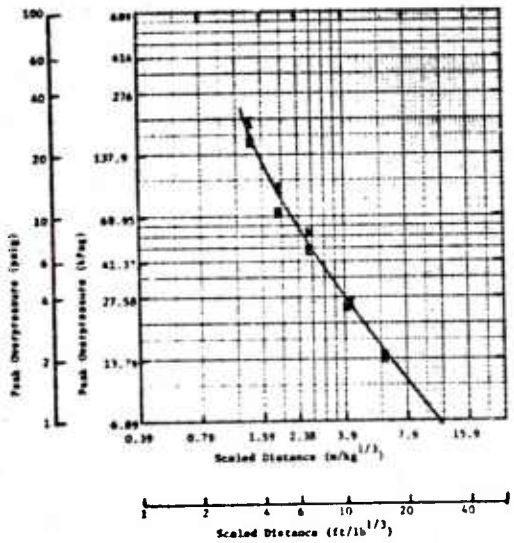


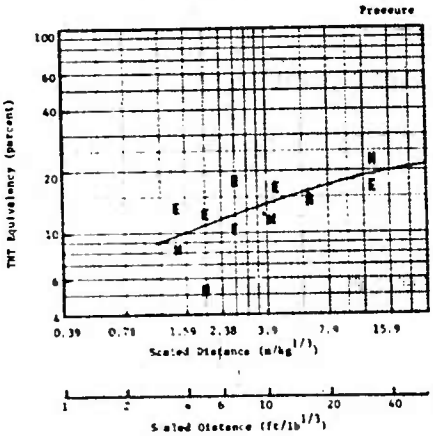


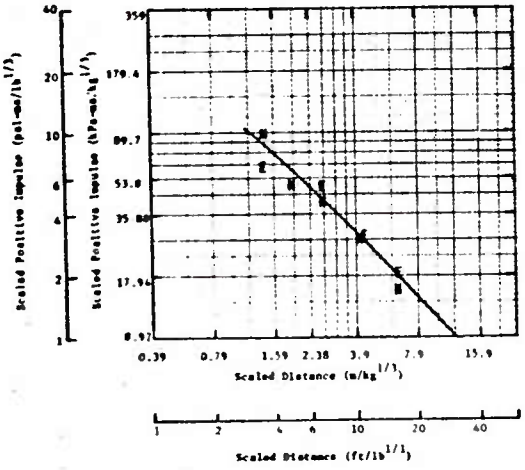


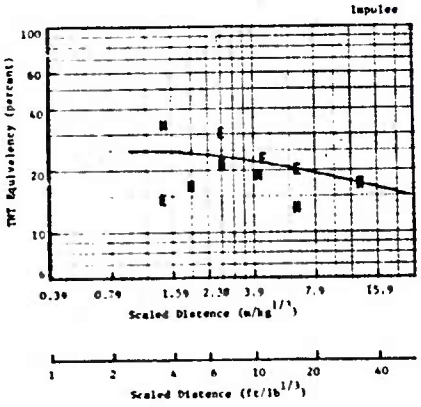


Airblast output for test 11 (M1)

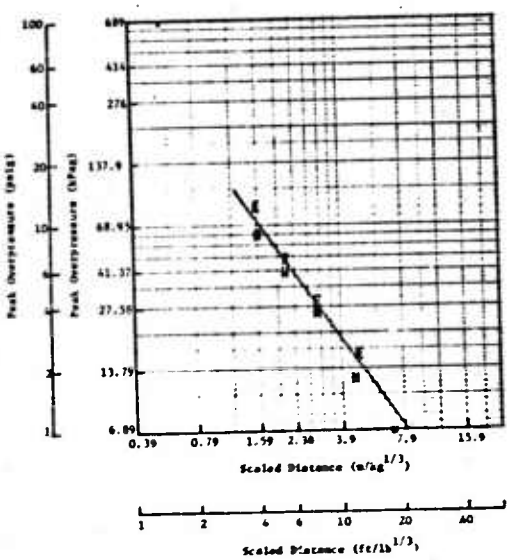


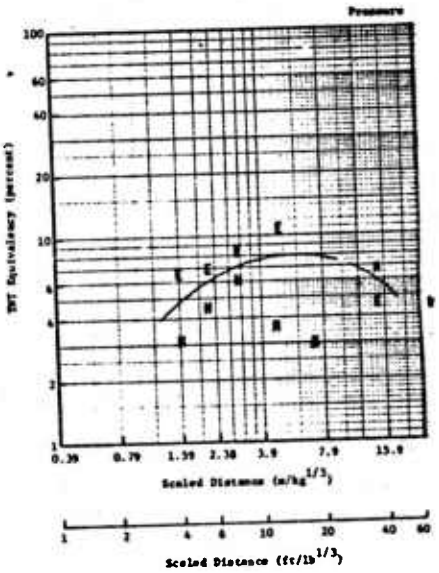


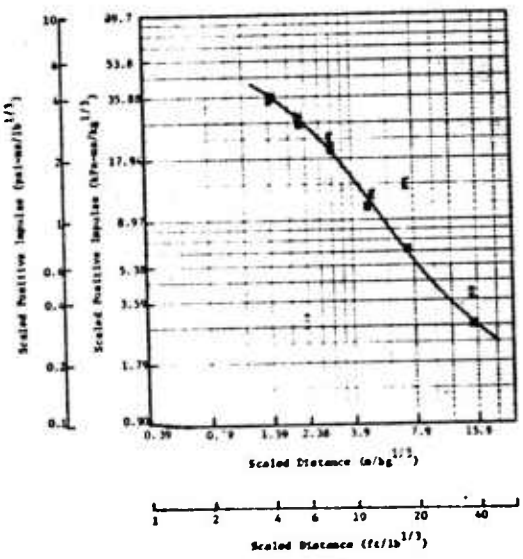


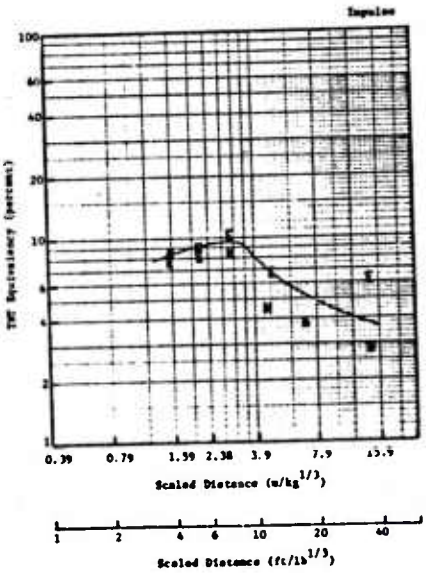


Airblast output for test 12 (M1)









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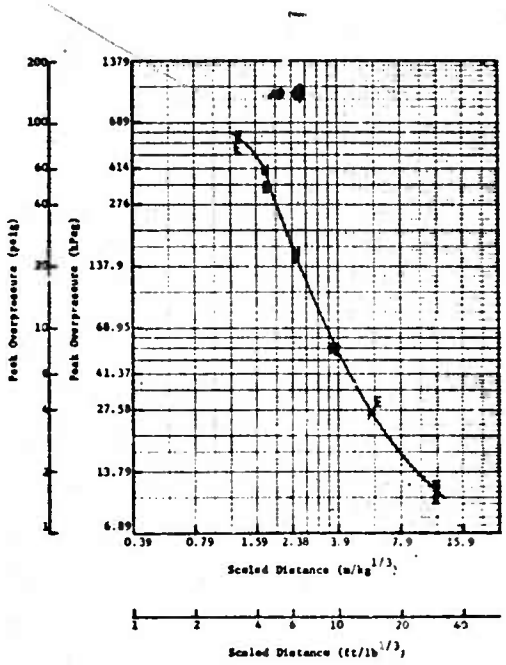

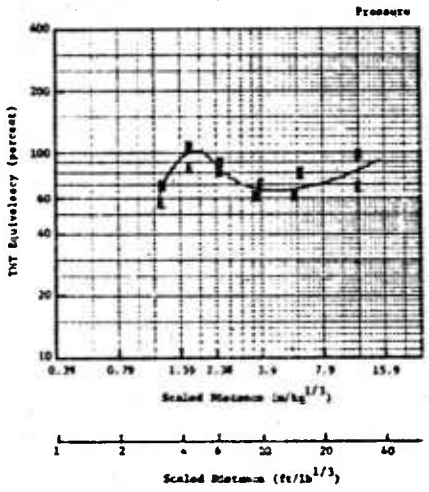

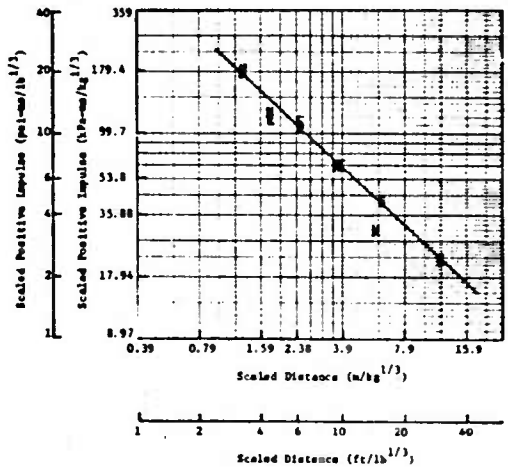

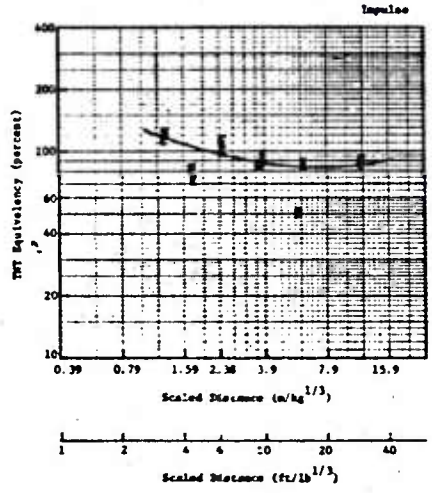

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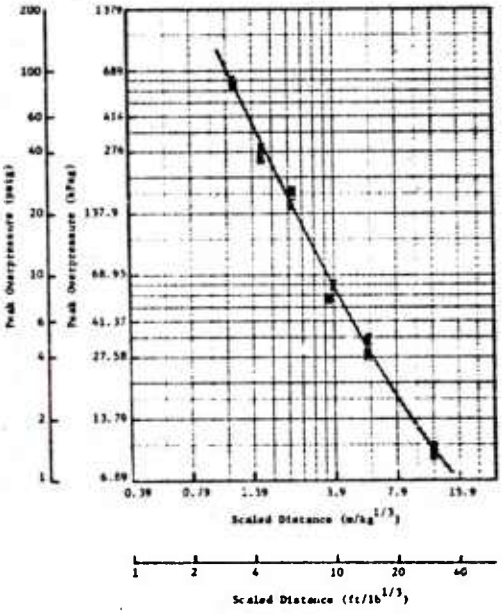

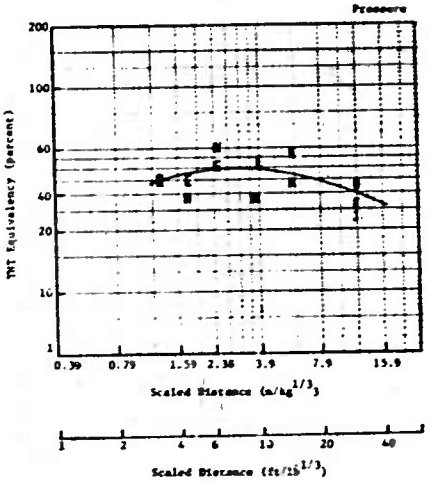

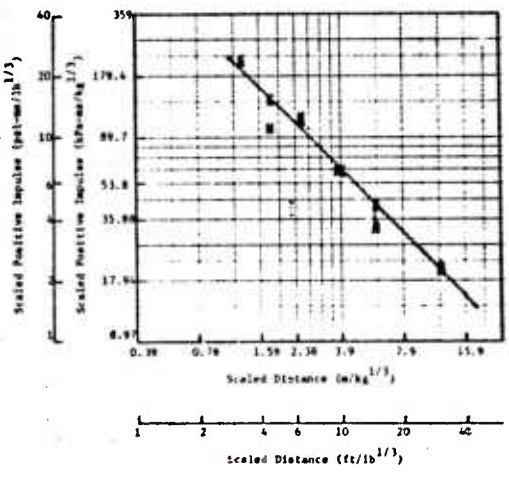

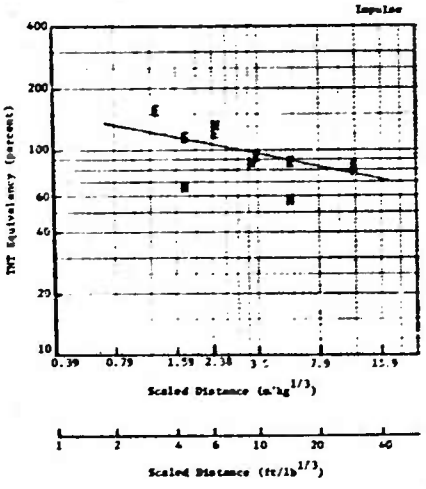

Airblast output for test 16 (M26)

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